





BSI Standards Publication

Reaction-to-fire tests — Heat release, smoke production and mass loss rate

Part 1: Heat release rate (cone calorimeter method) and smoke production rate (dynamic measurement)



National foreword

This British Standard is the UK implementation of ISO 5660-1:2015. It supersedes BS 476-15:1993, ISO 5660-1:1993 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee FSH/21/-/5, Reaction to fire tests - Heat release and smoke measurement.

A list of organizations represented on this committee can be obtained on request to its secretary.

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ISBN 978 0 580 77251 1 ICS 13.220.50

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 March 2015.

Amendments/corrigenda issued since publication

Date Text affected

INTERNATIONAL STANDARD

ISO 5660-1:2015 ISO 5660-1

Third edition 2015-03-15

Reaction-to-fire tests — Heat release, smoke production and mass loss rate —

Part 1:

Heat release rate (cone calorimeter method) and smoke production rate (dynamic measurement)

Essais de réaction au feu — Débit calorifique, taux de dégagement de fumée et taux de perte de masse —

Partie 1: Débit calorifique (méthode au calorimètre à cône) et taux de dégagement de fumée (mesurage dynamique)





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Published in Switzerland

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 92, *Fire safety*, Subcommittee SC 1, *Fire initiation and growth*.

This third edition of ISO 5660-1 cancels and replaces ISO 5660-1:2002 (second edition) and ISO 5660-2:2002 (first edition), which have been technically revised and merged.

ISO 5660 consists of the following parts, under the general title *Reaction-to-fire tests* — *Heat release, smoke production and mass loss rate*:

- Part 1: Heat release rate (cone calorimeter method) and smoke production rate (dynamic measurement)
- *Part 3: Guidance on measurement* [Technical Specification]

The following part is under preparation:

— Part 4: Measurement of heat release for determination of low levels of combustibility.

Reaction-to-fire tests — Heat release, smoke production and mass loss rate —

Part 1:

Heat release rate (cone calorimeter method) and smoke production rate (dynamic measurement)

1 Scope

This part of ISO 5660 specifies a method for assessing the heat release rate and dynamic smoke production rate of specimens exposed in the horizontal orientation to controlled levels of irradiance with an external igniter. The heat release rate is determined by measurement of the oxygen consumption derived from the oxygen concentration and the flow rate in the combustion product stream. The time to ignition (sustained flaming) is also measured in this test.

The dynamic smoke production rate is calculated from measurement of the attenuation of a laser light beam by the combustion product stream. Smoke obscuration is recorded for the entire test, regardless of whether the specimen is flaming or not.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 554, Standard atmospheres for conditioning and/or testing — Specifications

ISO 13943, Fire safety — Vocabulary

ISO 14697, Fire tests — Guidance on the choice of substrates for building products

3 Terms and definitions

For the purposes of this international standard, the terms and definitions given in ISO 13943 and the following apply.

3.1

essentially flat surface

surface whose irregularity from a plane does not exceed ± 1 mm

3.2

flashing

existence of flame on or over the surface of the specimen for periods of less than 1 s

3.3

ignition

onset of sustained flaming as defined in 3.10

3.4

irradiance

(at a point of a surface) quotient of the radiant flux incident on an infinitesimal element of surface containing the point, and the area of that element

Note 1 to entry: Convective heating is negligible in the horizontal specimen orientation. For this reason, the term "irradiance" is used instead of "heat flux" throughout this part of ISO 5660 as it best indicates the essentially radiative mode of heat transfer.

3.5

material

single substance or uniformly dispersed mixture

EXAMPLE Metal, stone, timber, concrete, mineral fibre and polymers

3.6

orientation

plane in which the exposed face of the specimen is located during testing, with either the vertical or horizontally face upwards

3.7

oxygen consumption principle

proportional relationship between the mass of oxygen consumed during combustion and the heat released

3.8

product

material, composite or assembly about which information is required

3.9

specimen

representative piece of the product which is to be tested together with any substrate or treatment

Note 1 to entry: For certain types of product, for example products that contain an air gap or joints, it may not be possible to prepare specimens that are representative of the end-use conditions (see <u>Clause 7</u>).

3 10

sustained flaming

existence of flame on or over the surface of the specimen for periods of over 10 s

3.11

transitory flaming

existence of flame on or over the surface of the specimen for periods of between 1 and 10 s

3.12

smoke obscuration

reduction, usually expressed as a percentage, in the intensity of light due to its passage through smoke

3.13

extinction coefficient

 $natural\ logarithm\ of\ the\ ratio\ of\ incident\ light\ intensity\ to\ transmitted\ light\ intensity,\ per\ unit\ light\ path\ length$

3.14

smoke production

integral of the smoke production rate over the time interval being considered

3.15

smoke production rate

product of the volumetric flow rate of smoke and the extinction coefficient of the smoke at the point of measurement

4 Symbols

See <u>Table 1</u>.

Table 1 — Symbols and their designations and units

Symbol	Designations	Unit
A	exposed surface area of specimen	m ²
A_{S}	initially exposed surface area of the specimen	m ²
С	orifice flow meter calibration constant	m ^{1/2} g ^{1/2} K ^{1/2}
D'	optical density	1
F	optical density calibration factor	m-1
$\Delta h_{\rm c}$	net heat of combustion	K J g ⁻¹
$\Delta h_{\rm c,eff}$	effective net heat of combustion	M J kg ⁻¹
I_0/I	ratio of incident light to transmitted light	1
k	linear Napierian absorption coefficient (commonly called extinction coefficient)	m-1
k_1	measured calibration extinction coefficient	m-1
k_2	calculated calibration extinction coefficient	m-1
k_{m}	measured extinction coefficient	m-1
L	light path length through smoke	m
m	mass of the specimen	g
Δm	total mass loss	g
$m_{ m f}$	mass of the specimen at the end of the test	g
$m_{\rm S}$	mass of the specimen at sustained flaming	g
$\dot{m}_{A,10-90}$	average mass loss rate per unit area between 10 % and 90 % of mass loss	g m ⁻² s ⁻¹
m_{10}	mass of the specimen at 10 % of total mass loss	g
m_{90}	mass of the specimen at 90 % of total mass loss	g
ṁ	mass loss rate of the specimen	g s-1
\dot{m}_e	mass flow rate in exhaust duct	kg s ⁻¹
M	molecular weight of the gases flowing through the exhaust duct	kg mol ⁻¹
Δр	orifice meter pressure differential	Pa
$P_{\rm S}$	smoke production rate	m ² s ⁻¹
$P_{S,A}$	smoke production rate normalized to the specimen area	s-1
\dot{q}	heat release rate	kW
$\dot{q}_{ ext{A}}$	heat release rate per unit area	kW m ⁻²
$\dot{q}_{ ext{A,max}}$	maximum value of the heat release rate per unit area	kW m⁻²
$\dot{q}_{\scriptscriptstyle m A,180}$	average heat release rate per unit area over the period starting at $t_{ m ig}$ and ending 180 s later	kW m ⁻²
$\dot{q}_{ ext{A,300}}$	average heat release rate per unit area over the period starting at $t_{\rm ig}$ and ending 300 s later	kW m⁻²
$Q_{ m A,tot}$	total heat released per unit area during the entire test	MJ m ^{−2}
r _o	stoichiometric oxygen/fuel mass ratio	1

Table 1 (continued)

Symbol	Designations	Unit
S	total smoke production	m ²
S_{A}	total smoke production per unit area	$m^2 m^{-2}$
$S_{A,1}$	total smoke production per unit area before ignition	m ² m ⁻²
$S_{A,2}$	total smoke production per unit area after ignition	$m^2 m^{-2}$
t	time	S
$t_{ m d}$	delay time of the oxygen analyser	S
$t_{ m ig}$	time to ignition (onset of sustained flaming)	S
Δt	sampling time interval	s
t_{10}	time at 10 % of total mass loss	S
t ₉₀	time at 90 % of total mass loss	s
$T_{\rm e}$	absolute temperature of gas at the orifice meter	K
$T_{\rm S}$	temperature of the smoke at the point of measurement	K
\dot{V}_{s}	volume flow rate of smoke at the point of measurement	m ³ s ⁻¹
X ₀₂	oxygen analyser reading, mole fraction of oxygen	1
X^0_{02}	initial value of oxygen analyser reading	1
X ¹ 02	oxygen analyser reading, before delay time correction	1
ρ	density	kg m ^{−3}
σ	specific extinction area	$m^2 kg^{-1}$

NOTE Detailed discussion of some of these parameters and their units is given in reference[11].

5 Principle

The test method is based on the observation that, generally, the net heat of combustion is proportional to the amount of oxygen required for combustion. The relationship is that approximately 13.1×10^3 kJ of heat are released per kilogram of oxygen consumed. Specimens in the test are burned under ambient air conditions, while being subjected to a predetermined external irradiance within the range of 0 kW m⁻² to 75 kW m⁻² and measurements are made of oxygen concentrations and exhaust gas flow rates.

This test method is used to assess the contribution that the product under test can make to the rate of evolution of heat during its involvement in fire. These properties are determined on small representative specimens.

The principle of the smoke measurement is based on the observation that, generally, the intensity of light that is transmitted through a volume of combustion products is an exponentially decreasing function of distance. This is commonly referred to as Bouguer's law. Specimens in the test are burned in ambient air conditions, while being subjected to a predetermined external irradiance within the range 0 kW $\,\mathrm{m}^{-2}$ to 75 kW $\,\mathrm{m}^{-2}$ and measurements are made of smoke obscuration, exhaust gas flow rate, and mass loss rate of the specimen. Smoke obscuration is measured as the fraction of laser light intensity that is transmitted through the smoke in the exhaust duct. This fraction is used to calculate the extinction coefficient according to Bouguer's law. The test results are reported in terms of smoke production and smoke production rate-both normalized to exposed specimen surface area. Smoke production rate is calculated as the product of the extinction coefficient and the volumetric flow rate of the smoke in the exhaust duct. Smoke production is calculated by numerical integration of the smoke production rate over the time interval being considered. The variables reported are normalized to area because smoke production is proportional to area.

The test method is used to assess the contribution that the product under test can make to the rate of evolution of smoke and to the amount of smoke produced during its involvement in a well-ventilated fire. These properties are once again determined on small representative specimens.

6 Apparatus

6.1 General

A schematic representation of the apparatus required for this part of ISO 5660 is given in <u>Figure 1</u>. The individual components are described in detail in <u>6.2</u> to <u>6.19</u>.

Carbon Monoxide and Carbon dioxide measurements can optionally, and additionally, be made and used in the calculation of the heat release rate. The apparatus, procedures and calculation methods described in $\underline{\mathsf{Annex}\;\mathsf{G}}$ are then applicable.

With minor modifications to the apparatus, specimens may be tested in the vertical orientation. <u>Annex E</u> gives guidance on these modifications.

6.2 Cone-shaped radiant electrical heater

The active element of the heater shall consist of an electrical heater rod, capable of delivering 5 000 W at the operating voltage, tightly wound into the shape of a truncated cone (see Figure 2). The heater shall be encased on the outside with a double-wall stainless steel cone, filled with a refractory fibre blanket of nominal thickness 13 mm and nominal density 100 kg m^{-3} . The irradiance from the heater shall be maintained at a preset level by controlling the average temperature of three thermocouples (type K stainless steel sheathed thermocouples have proved suitable but Inconel or other high performance materials are also acceptable), symmetrically disposed and in contact with, but not welded to, the heater element (see Figure 2). Either 3,0 mm outside diameter sheathed thermocouples with exposed hot junction or 1,0 mm to 1,6 mm outside diameter sheathed thermocouples with unexposed hot junction shall be used. The heater shall be capable of producing irradiance on the surface of the specimen of up to 75 kW m⁻². The irradiance shall be uniform within the central 50 mm x 50 mm area of the exposed specimen surface, to within ± 2 %. for an irradiance of 50 kW m⁻²

6.3 Radiation shield

The cone heater shall be provided with a removable radiation shield to protect the specimen from the irradiance prior to the start of a test. The shield shall be made of non-combustible material, with a total thickness not exceeding 12 mm. The shield shall be one of the following, either:

- a) water cooled and coated with a durable matt black finish of surface emissivity $\varepsilon = 0.95 \pm 0.05$; or
- b) not water-cooled, which may be either metal with a reflective top surface, or metal with a ceramic top surface, or ceramic, in order to minimize radiation transfer.

The shield shall be equipped with a handle or other suitable means for quick insertion and removal. The cone heater base plate shall be equipped with a mechanism for moving the shield into position.

6.4 Irradiance control

The irradiance control system shall be properly tuned so that it maintains the average temperature of the heater thermocouples during the calibration described in 10.1.2 at the preset level to within ± 10 °C.

6.5 Weighing device

The weighing device shall have a resolution of 0,1 g and an accuracy of \pm 0,3 g or better, measured according to the calibration procedure described in Clause 10.2.2. The weighing device shall be capable of measuring the mass of specimens of at least 500 g. The weighing device shall have a 10 % to 90 % response time of 4 s or less, as determined according to the calibration described in 10.1.3. The output

of the weighing device shall not drift by more than 1 g over a 30 min period, as determined with the calibration described in <u>Clause 10.1.4</u>.

6.6 Specimen holder

The specimen holder is shown in Figure 3. The specimen holder shall have the shape of a square pan with an opening of (106 ± 1) mm x (106 ± 1) mm at the top, and a depth of (25 ± 1) mm. The holder shall be constructed of stainless steel with a thickness of $(2,4 \pm 0,15)$ mm. It shall include a handle to facilitate insertion and removal, and a mechanism to ensure central location of the specimen under the heater and proper alignment with the weighing device. The bottom of the holder shall be lined with a layer of low density (nominal density 65 kg m^{-3}) ceramic fibre blanket with a thickness of at least 13 mm. The distance between the bottom surface of the cone heater and the top of the specimen shall be adjusted to be (25 ± 1) mm, except for dimensionally unstable materials for which the distance shall be (60 ± 1) mm (see Clause 7.5).

6.7 Retainer frame

The frame shall be constructed of stainless steel with a thickness of (1.9 ± 0.1) mm in the shape of a box with an inside dimension of each side (111 ± 1) mm and a height of (54 ± 1) mm. The opening for the specimen face shall be (94.0 ± 0.5) mm square as shown in Figure 4. The retainer frame shall have an appropriate means to secure it to the specimen holder with a specimen in position.

6.8 Exhaust gas system with flow measuring instrumentation

The exhaust gas system shall consist of a centrifugal exhaust fan rated for the operating temperatures, a hood, intake and exhaust ducts for the fan, and an orifice plate flow meter (see Figure 5). The distance between the bottom of the hood and the specimen surface shall be (210 \pm 50mm). The exhaust system shall be capable of developing flows up to 0,035 m³ s⁻¹, under standard conditions of temperature and pressure. The recommended location of the fan is indicated in Figure 5. As an alternative, it is acceptable to locate the fan further downstream and to have the measuring orifice before the fan, provided that the requirements described in the remainder of this clause are fulfilled.

A restrictive orifice with an internal diameter of (57 ± 3) mm shall be located between the hood and the duct to promote mixing.

A gas sampling ring probe shall be located in the fan intake duct for gas sampling, (685 \pm 15) mm from the hood (see Figure 5). The gas sampling ring probe shall contain 12 small holes with a diameter of (2,2 \pm 0,1) mm, to average the stream composition, with the holes facing away from the flow to avoid clogging with soot.

The temperature of the gas stream shall be measured using a 1,0 mm to 1,6 mm outside diameter sheathed-junction thermocouple or a 3 mm outside diameter exposed-junction thermocouple positioned in the exhaust stack on the centreline and (100 ± 5) mm upstream from the measuring orifice plate.

The flow rate shall be determined by measuring the differential pressure across a sharp edge orifice [internal diameter (57 \pm 3) mm, thickness (1,6 \pm 0,3) mm] in the exhaust stack, at least 350 mm downstream from the fan, if the latter is located as shown in Figure 5. If the fan is located further downstream than indicated in Figure 5, it is acceptable to locate the orifice plate between the gas sampling ring probe and the fan. However, in that case the length of the straight duct section on both sides of the orifice plate shall be at least 350 mm.

6.9 Gas sampling apparatus

Gas sampling apparatus incorporates a pump, filters to prevent entry of soot, facilities for removal of the moisture, a by-pass system set to divert all flow except that required for the gas analysers, a further moisture trap and a trap for CO_2 removal.

A schematic view of an example of the gas sampling apparatus is shown in <u>Figure 6</u>. Other arrangements which satisfy the requirements may be used. The transport delay time of the oxygen analyser, t_d , shall be determined according to <u>10.1.5</u>, and shall not exceed 60s.

NOTE If an (optional) CO_2 analyser is used, the formulae used to calculate the heat release rate can be different from those for the standard case (see <u>Clause 12</u> and <u>Annex G</u>).

6.10 Ignition circuit

External ignition is accomplished by a spark plug powered from a 10 kV transformer or spark igniter. The spark plug shall have a gap of $(3,0\pm0,5)$ mm. The electrode length and location of the spark plug shall be such that the spark gap is located (13 ± 2) mm above the centre of the specimen, except for dimensionally unstable materials for which the distance is (48 ± 2) mm (see Clause 7.5).

6.11 Ignition timer

The ignition timer shall be capable of recording elapsed time to the nearest second and shall be accurate to within 1 s in one hour.

6.12 Oxygen analyser

The oxygen analyser shall be of the paramagnetic type, with a range of at least 0 % oxygen to 25 % oxygen. The analyser shall exhibit a drift of not more than 50 μ l/l of oxygen over a period of 30 minutes, and a noise of not more than 50 μ l/l of oxygen during this 30-min period, as measured according to 10.1.6. Since oxygen analysers are sensitive to stream pressures, the stream pressure shall be regulated (upstream of the analyser) to minimize flow fluctuations, and the readings from the analyser compensated with an absolute pressure transducer to allow for atmospheric pressure variations. The analyser and the absolute pressure transducer shall be located in an isothermal environment. The temperature of the environment shall be maintained to within 2 °C of a preset value between 30 °C and 70 °C. The oxygen analyser shall have a 10 % to 90 % of full-scale response time of less than 12 s, as measured according to 10.1.5.

6.13 Heat flux meters

The working heat flux meter shall be used to calibrate the heater (see 10.2.5). It shall be positioned at a location equivalent to the centre of the specimen face during this calibration.

This heat flux meter shall be of the Schmidt-Boelter (thermopile) type with a design range of (100 \pm 10) kW m $^{-2}$. The target receiving the heat shall be flat, circular, of approximately 12,5 mm in diameter and coated with a durable matt black finish of surface emissivity ϵ = 0,95 \pm 0,05. The body of the heat flux meter shall be water-cooled. A cooling temperature which would cause condensation of water on the target surface of the heat flux meter shall not be used.

Radiation shall not pass through any window before reaching the target. The instrument shall be robust, simple to set up and use, and stable in calibration. The instrument shall have a repeatability to within \pm 0,5 %.

The calibration of the working heat flux meter shall be checked according to $\underline{10.4.1}$, by comparison with two instruments of the same type as the working heat flux meter and of similar range held as reference standards and not used for any other purpose (see $\underline{\text{Annex } F}$). One of the reference standards shall be fully calibrated at a standardizing laboratory at yearly intervals.

6.14 Calibration burner

The calibration burner shall be constructed from tube with a square or circular orifice with an area of $(500 \pm 100) \, \text{mm}^2$ covered with wire gauze through which the methane diffuses. The tube is packed with refractory fibre to improve uniformity of flow. The calibration burner is suitably connected to a metered supply of methane of at least 99,5 % purity. The accuracy of the flow meter or controller shall be $\pm 3 \, \%$ of

the readout corresponding to a heat release rate of 5 kW. The accuracy verification shall be performed according to <u>Clause 10.4.3</u>.

6.15 Data collection and analysis system

The data collection and analysis system shall have facilities for recording the output from the oxygen analyser, the orifice meter, the thermocouples and the weighing device. The data collection system shall have an accuracy corresponding to at least 50 μ l/l of oxygen for the oxygen channel, 0,5 °C for the temperature measuring channels, 0,01 % of full-scale instrument output for all other instrument channels, and at least 0,1 % for time. The system shall be capable of recording data every second. The system shall be capable of storing a minimum of 720 data points per parameter. The raw data recorded for each test shall be stored so that it can be recovered and used to check the accuracy of the software.

6.16 Optional side screens

For operational or safety reasons, it is permitted to guard the heater and sample holder with side screens. However, it shall be demonstrated that the presence of the screens does not affect the ignition time and heat release rate measurements according to the procedure described in 10.1.7.

If the screens form an enclosure, the attention is drawn to the fact that there is a possible explosion hazard when the instrument is not operated under conditions prescribed by this part of ISO 5660, in particular for experiments in oxygen enriched atmosphere. If an explosion hazard exists, proper precautions shall be taken to protect the operator, e.g. by installing an explosion vent facing away from the operator.

6.17 Smoke obscuration measuring system

For measuring the attenuation of laser light in the exhaust duct; the system comprises a helium-neon laser (between 0,5 mW and 2 mW, polarized), silicon photodiodes as main beam and reference detectors, and appropriate electronics to derive the extinction coefficient and to set the zero reading. The meter shall be located horizontally (111 ± 1) mm downstream of the gas sampling ring. Two small diameter tubes welded onto each side of the exhaust duct serve as part of the light baffling for the purging air and also allow for any smoke that may enter, despite the purge flow, to be deposited on the tube walls before reaching the optical elements. One acceptable arrangement of a smoke measuring system is shown in Figure 7.

NOTE Experimental work has been performed with systems using a white light source with collimating optics[12]. Such systems have been shown to yield generally similar results [17-19] but not under all conditions[20]. Theoretical predictions [21] have been verified experimentally. White light systems may be used if shown to have an equivalent accuracy.

6.18 Smoke system thermocouple

To measure the temperature of the gas stream near the smoke meter. This temperature shall be measured using a 1,0mm to 1,6mm outside diameter unearthed sheathed-junction thermocouple or a 3 mm outside diameter exposed-junction thermocouple positioned in the exhaust stack on the centre line and 50 mm downstream from the smoke meter, as shown in Figure 5.

6.19 Optical filters

To calibrate the smoke obscuration measuring system. Two glass neutral density dispersion filters^[22], accurately calibrated at the laser wavelength of 632,8 nm, are required. The filters used shall not be of the coated type because these filters can give rise to interference effects with laser light and can deteriorate with time. The filters shall have nominal optical densities of 0,3 and 0,8. Corresponding values of extinction coefficient, k, are obtained from the formula:

$$k = (2,303 D') L^{-1}$$

7 Suitability of a product for testing

7.1 Surface characteristics

A product having one of the following properties is suitable for testing:

- a) an essentially flat exposed surface;
- b) a surface irregularity which is evenly distributed over the exposed surface provided that
 - 1) at least 50% of the surface of a representative 100mm square area lies within a depth of 10 mm from a plane taken across the highest points on the exposed surface,

or

2) for surfaces containing cracks, fissures or holes not exceeding 8mm in width nor 10mm in depth, the total area of such cracks, fissures or holes at the surface does not exceed 30 % of a representative 100mm square area of the exposed surface.

When an exposed surface does not meet the requirements of either <u>7.1</u> a) or <u>7.1</u> b), the product shall be tested in a modified form complying as nearly as possible with the requirements given in <u>7.1</u>. The test report shall state that the product has been tested in a modified form, and clearly describe the modification.

7.2 Asymmetrical products

A product submitted for this test can have faces which differ or can contain laminations of different materials arranged in a different order in relation to the two faces. If either of the faces can be exposed in use within a room, cavity or void, then both faces shall be tested.

7.3 Materials of short burning time

For specimens of short burning time (3 min or less), the heat release rate measurements shall be taken at not more than 2 s intervals. For longer burning times, 5 s intervals may be used.

7.4 Composite specimens

Composite specimens are suitable for testing, provided they are prepared as specified in <u>8.3</u> and are exposed in a manner typical of end use conditions.

7.5 Dimensionally unstable materials

Samples that intumesce or deform so that they contact the spark plug prior to ignition, or the underside of the cone heater after ignition, shall be tested with the separation of 60 mm between the base plate of the cone heater and the upper surface of the specimen. In this case the heater calibration (see 10.2.5) shall be performed with the heat flux meter positioned 60 mm below the cone heater base plate. It shall be stressed that the time to ignition measured with this separation is not comparable to that measured with the separation of 25 mm.

Other dimensionally unstable products, for example products that warp or shrink during testing, shall be restrained against excessive movement. This shall be accomplished with 4 tie wires, as described below. Metal wires of $(1,0 \pm 0,1)$ mm diameter and at least 350 mm long shall be used. The sample shall be prepared in the standard way as described in Clause 8. A tie wire is then looped around the sample holder and retainer frame assembly, so that it is parallel to and approximately 20 mm away from one of the 4 sides of the assembly. The ends of the wire are twisted together such that the wire is pulled firmly against the retainer frame. Excess wire is trimmed from the twisted section before testing. The 3 remaining wires shall be fitted around the specimen holder and retainer frame assembly in a similar manner, parallel to the three remaining sides.

Materials that distort so extensively that they cannot be held by 4 wires should be tested using the fine wire grid made of (0.8 ± 0.1) mm with wire spacing of (20 ± 2) mm shown in Figure 8.

Materials that intumesce in a fluid phase such that molten materials overflows the edge frame or seep between the edge frame and the specimen holder invalidate the test. Therefore, such materials should be tested without the edge frame and should be housed in 0,1 mm thick aluminium tray wrappings which extends 10mm above the top edge of the test specimen.

7.6 Materials that require testing under compression

Materials, such as fibres, which need to be both physically restrained or compressed to be tested at installed densities should be tested in a wire cage structure made of $(1,0 \pm 0,1)$ mm steel wire with (9 ± 1) mm spacing as shown in Figure 9, which provides appropriate artificial boundaries to enable the materials to be tested (see Clause 8.3.3).

8 Specimen construction and preparation

8.1 Specimens

- **8.1.1** Unless otherwise specified, three specimens shall be tested at each level of irradiance selected and for each different exposed surface.
- **8.1.2** The specimens shall be representative of the product and shall be square with sides measuring 100_{-2}^{0} mm.
- **8.1.3** Products with normal thickness of 50 mm or less shall be tested using their full thickness.
- **8.1.4** For products with a normal thickness of greater than 50 mm, the requisite specimens shall be obtained by cutting away the unexposed face to reduce the thickness to 50 mm.
- **8.1.5** When cutting specimens from products with irregular surfaces, the highest point on the surface shall be arranged to occur at the centre of the specimen.
- **8.1.6** Assemblies shall be tested as specified in <u>8.1.3</u> or <u>8.1.4</u> as appropriate. However, where thin materials or composites are used in the fabrication of an assembly, the nature of any underlying construction, including any air gaps, can significantly affect the ignition and burning characteristics of the exposed surface.

The influence of the underlying layers shall be understood and care taken to ensure that the test result obtained on any assembly is relevant to its use in practice.

When the product is a material or composite which would normally be attached to a well-defined substrate, it shall be tested in conjunction with that substrate using the recommended fixing technique, for example bonded with the appropriate adhesive or mechanically fixed. In the absence of a unique or well-defined substrate, an appropriate substrate for testing shall be selected in accordance with ISO 14697.

8.1.7 Products that are thinner than 6mm shall be tested with a substrate representative of end-use conditions, such that the total specimen thickness is 6 mm or more. However, some products thinner than 6 mm could be tested in specific cases and placed directly above refractory fibre blanket. This shall be mentioned in test report.

8.2 Conditioning of specimens

Before the test, specimens shall be conditioned to constant mass at a temperature of (23 ± 2) °C, and a relative humidity of (50 ± 5) % in accordance with ISO 554.

Constant mass is considered to be reached when two successive weighing operations, carried out at an interval of 24 h, do not differ by more than 0,1 % of the mass of the test piece or 0,1 g, whichever is the greater.

Materials such as polyamides, which require more than one week in conditioning to reach equilibrium may be tested after conditioning in accordance with ISO 291[1]. This period shall be not less than one week, and shall be described in the test report.

8.3 Preparation

8.3.1 Specimen wrapping

A conditioned specimen shall be wrapped in a single layer of aluminium foil, of 0,025 mm to 0,04 mm thickness, with the shiny side towards the specimen. The aluminium foil shall be pre-cut to a size to cover the bottom and sides of the specimen and extend 3mm or more beyond the upper surface of the specimen. The specimen shall be placed in the middle of the foil and the bottom and sides shall be wrapped. The excess foil above the top surface shall be cut if necessary so that it does not extend more than 3mm above the top surface of the specimen. The excess foil at the corners shall be folded around the corners to form a seal around the top surface of the specimen. After wrapping, the wrapped specimen shall be placed in the specimen holder and covered by a retainer frame. No aluminium foil shall be visible after the procedure is completed.

For soft specimens, a dummy specimen having the same thickness as the specimen to be tested may be used to pre-shape the aluminium foil.

8.3.2 Specimen preparation

All specimens (other than those that intumesce in a fluid phase or those that require testing under compressing in specimen cages, as in <u>Clause 8.3.3</u>) shall be tested with the retainer frame shown in <u>Figure 4</u>. The following steps shall be taken to prepare a specimen for testing:

- a) put the retainer frame on a flat surface facing down;
- b) insert the foil-wrapped specimen into the frame with the exposed surface facing down;
- c) put layers of refractory fibre blanket (nominal thickness 13mm, nominal density 65kg/m³) on top until at least one full layer, and not more than two layers extend above the rim of the frame;
- d) fit the sample holder into the frame on top of the refractory fibre and press down; and
- e) Secure the retainer frame to the specimen holder and re-invert the holder ready for testing

8.3.3 Preparing specimens of materials that require testing under compression

- a) Construct the specimen test cage shown in Figure 9 by forming a 241 mm \times 101 mm sheet of steel mesh, constructed from (1,0 \pm 0,1) mm steel wire with (9 \pm 1) mm spacing wire separation, around a solid template 10 mm \times 100 mm \times 100 mm.
- b) Remove the template and then pack the test material into the specimen cage such that the density is that used in the installed use.
- c) Test the specimen as described in 8.3.1 but do not use the retainer frame

9 Test environment

The apparatus shall be located in an essentially draught-free environment in an atmosphere of relative humidity of between 20 % and 80 % and a temperature between 15 °C and 30 °C.

10 Calibration

10.1 Preliminary calibrations

10.1.1 General

The calibrations in this section, except for that in <u>Clause 10.1.7</u>, shall be performed before conducting experiments, when commissioning a Cone calorimeter; or after maintenance, repair or replacement of the heater assembly or irradiance control system (10.1.2), the weighing device (10.1.3 and 10.1.4), the oxygen analyser or other major components of the gas analysis system (10.1.5 and 10.1.6). The calibration tests to determine the effect of side screens in 10.1.7 are conducted at the time the screens are installed. For a new instrument that is delivered with side screens, this shall be done by the manufacturer.

10.1.2 Irradiance control system response characteristics

Turn on power to the cone heater and the exhaust fan. Set an irradiance of (50 ± 1) kW m⁻², and an exhaust flow rate of (0.024 ± 0.002) m³ s⁻¹. After reaching equilibrium of the heater, record the average heater temperature. Test a specimen of black poly(methyl-methacrylate) (PMMA), without the retainer frame, according to the procedure in <u>Clause 11</u>. The PMMA specimen shall have a thickness of at least 6 mm. The validity of the test shall be checked against the expected average heat release rate recorded over the first three minutes following ignition for the specific batch of PMMA which should be provided by the supplier. During the test, record the average heater temperature at 5 s intervals.

10.1.3 Weighing device response time

The cone heater shall not be turned on for this calibration. Place an empty specimen holder with a (500 ± 25) g mass on the weighing device. The mass accounts for the retainer frame, which is not used during this calibration. Measure the weighing device output, and mechanically or electronically adjust the value to zero. Gently add a second mass of (250 ± 25) g on the holder and record the weighing device output. After equilibrium is reached, gently remove the mass from the holder, and again record the weighing device output. Determine the response time of the weighing device as the average of the times for the weighing device output to change from 10% to 90% of its ultimate deflection.

10.1.4 Weighing device output drift

Set the height of the cone heater to the same position as when testing a specimen with the retainer frame. Place a thermal barrier on the weighing device. Turn on power to the exhaust fan and cone heater. Set an exhaust flow rate of (0.024 ± 0.002) m³ s⁻¹ and an irradiance of (50 ± 1) kW m⁻². After reaching equilibrium of the heater temperature, remove the thermal barrier and place an empty specimen holder with a (500 ± 25) g mass on the weighing device. The mass accounts for the retainer frame, which is not used during this calibration. After equilibrium is reached, with an interval of at least 5 s measure the weighing device output and mechanically or electronically adjust the value to zero. Gently add a second mass of (250 ± 25) g on the specimen holder. After equilibrium is reached, record the weighing device output. After 30 min, record the weighing device output. Calculate the drift of the weighing device output as the absolute value of the difference between the mean of 12 initial and 12 final values.

10.1.5 Oxygen analyser delay and response times

The cone heater shall not be turned on for this calibration. Turn on the exhaust fan, and set an exhaust flow rate of (0.024 ± 0.002) m³ s⁻¹. Determine the delay time of the oxygen analyser by delivering a methane flow rate equivalent to (5 ± 0.5) kW to the calibration burner. Light the burner outside the hood and allow the flame to stabilize. Quickly introduce the burner underneath the hood, and leave the burner in position for 3 min. Then, remove the burner from underneath the hood and turn off the methane supply. Record the output of the analyser from the moment of insertion of the burner underneath the hood, until 3 min after removal of the burner. The turn-on delay is the time difference between insertion of the burner and the oxygen reading reaching 50 % of its ultimate deflection. Calculate the turn-off

delay similarly. The delay time t_d is the average of at least three turn-on and turn-off delays. The oxygen concentration at a given time shall be taken as the concentration registered after the time interval t_d .

The response time of the oxygen analyser is calculated as the average for the turn-on and turn-off experiments of the time for the oxygen analyser output to change from 10 % to 90 % of its ultimate deflection.

The ultimate deflection is taken as the average oxygen concentration between 1 min and 3 min after insertion (or removal) of the burner.

10.1.6 Oxygen analyser output noise and drift

The cone heater shall not be turned on for this calibration. Turn on the exhaust fan, and set an exhaust flow rate of $(0,024 \pm 0,002)$ m³ s⁻¹. Feed the oxygen analyser with oxygen-free nitrogen gas. After 60 min, switch to dried ambient air from the exhaust duct at the normal flow rate and pressure as for the sample gases. After reaching equilibrium, adjust the oxygen analyser output to $(20,95 \pm 0,01)$ %. Start recording the oxygen analyser output at a maximum of 5 s intervals for a period of 30 min. Determine the drift by use of a least squares fitting procedure to fit a straight line through the data points. For the straight line fit, the absolute value of the difference between the reading at 0 and at 30 min represents the short-term drift. Determine the noise by computing the root-mean-square deviation around the linear trend line according to the following formula:

$$rms = \sqrt{\frac{\sum_{i=1}^{n} x_i^2}{n}} \tag{1}$$

where x_i is the absolute difference between the data point and the linear trend line.

Record this rms noise value in terms of $\mu l/l$ of oxygen.

10.1.7 Effect of side screens

To evaluate the effect of side screens on the test results, six specimens of black poly(methyl methacrylate) (PMMA) of thickness between 17 and 26 mm shall be tested at (50 \pm 1) kW m⁻² without the retainer frame, according to the procedure described in Clause 11. The first three tests shall be conducted with the screens removed, the remaining three tests with the screens in place. The screens are permitted if the differences between the average values of t_{ig} , $\dot{q}_{A,180}$, and $\dot{q}_{A,max}$ for the two test series are found to be statistically insignificant according to a two-sided t-test at a significance level of 5 %. This t-test shall be performed for the three variables (t_{ig} , $\dot{q}_{A,180}$, and $\dot{q}_{A,max}$) according to the following procedure.

a) for the two series of three tests, calculate the averages via

$$\overline{x} = \frac{\sum_{i=1}^{3} x_i}{3} \tag{2}$$

and

$$\overline{y} = \frac{\sum_{i=1}^{3} y_i}{3} \tag{3}$$

b) calculate the pooled standard deviation s_{p_i} from

$$s_{p} = \sqrt{\frac{\sum_{i=1}^{3} x_{i}(x_{i} - \overline{x})^{2} + \sum_{i=1}^{3} (y_{i} - \overline{y})^{2}}{4}}$$
(4)

c) calculate the t-test statistic as

$$t_s = \left| \frac{\overline{x} - \overline{y}}{0,8165 \, s_p} \right| \tag{5}$$

The *t*-test is successful if the value of the test statistic does not exceed 2,776, or if the two averages are equal.

10.2 Operating calibrations

10.2.1 General

The following calibrations shall be performed at the start of testing each day, in the order given below. The heater calibration shall also be performed when changing to a different irradiance level.

10.2.2 Weighing device accuracy

The weighing device shall be calibrated with standard weight pieces in the range of the test specimen mass. The cone heater shall be turned off and the apparatus shall be cooled down to ambient temperature before this calibration is performed. Place an empty specimen holder with a (500 ± 25) g mass on the weighing device. The mass accounts for the retainer frame, which is not used during this calibration. Measure the weighing device output, and mechanically or electronically adjust the value to zero. Gently add a mass between 50 g and 200 g on the holder and measure the weighing device output after it reaches a steady value. Repeat this procedure at least four times after adding masses of the same range. At the end of the calibration, the total mass on the holder shall be at least 500 g. The accuracy of the weighing device is determined as the maximum difference between the mass and the weighing device output recorded during the calibration. It shall be better than 0,3 g.

10.2.3 Oxygen analyser

Zero and calibrate the oxygen analyser. This calibration may be performed with the cone heater operating or not, but shall not be performed during heater warm-up. Turn on the exhaust fan, and set an exhaust flow rate of (0.024 ± 0.002) m³ s⁻¹. For zeroing, feed the analyser with oxygen-free nitrogen gas, with the same flow rate and pressure as for the sample gases. Adjust the analyser response to (0.00 ± 0.01) %. Calibration shall be similarly achieved using dried ambient air and adjusting for a response of (20.95 ± 0.01) %. Carefully monitor analyser flow rates and set to be equal to the flow rate used when testing specimens. After each specimen has been tested, ensure that a response level of (20.95 ± 0.01) % is obtained using dried ambient air.

10.2.4 Heat release rate calibration

Perform a heat release rate calibration to determine the orifice constant C. This calibration shall be performed with the cone heater operating or not, but shall not be performed during heater warm-up. Turn on the exhaust fan, and set an exhaust flow rate of (0.024 ± 0.002) m³ s⁻¹. Start collecting baseline data at 5s intervals for a period of at least 1-min. Introduce methane into the calibration burner using a calibrated flow meter or controller at a flow rate corresponding to $\dot{q}_b = (5 \pm 0.5)$ kW based on the net heat of combustion of methane (50.0×10^3) kJ kg⁻¹. Allow the methane to burn at a constant rate for 3 min then collect data at 5s intervals over a 3-min period. Calculate the orifice constant C according to Formula (6) in Clause 12, using averages over the three-min period of the measured values of \dot{q}_b , T_e , ΔP , and X_{02} . X^0_{02} is determined as the average of the oxygen analyser output measured during the 1-min baseline measurements.

An alternate procedure for performing this calibration consists of burning a suitable liquid fuel (e.g. ethanol) in a special pan that is placed on the weighing device. The average theoretical heat release rate is then obtained as the total mass of fuel burnt multiplied by the net heat of combustion of the fuel, and divided by the duration of flaming.

10.2.5 Heater calibration

At the start of testing each day, or when changing to a different irradiance level, adjust the irradiance control system so that the conical heater produces the required irradiance to within ± 2 %, as measured by the heat flux meter. No specimen or specimen holder shall be used when the heat flux meter is inserted into the calibration position. Operate the cone heater for at least 10 min when stable at set point.

10.3 Smoke meter calibration

10.3.1 Calibration with neutral density filters

The smoke meter shall be calibrated to read correctly (k to within 0,1 m⁻¹) for the two neutral density filters specified in 6.19, and 100 % transmission. This neutral density filter calibration will be performed at least every year or upon reassembling of the optics after cleaning and maintenance.

10.3.2 Calibration before test

Immediately before each test, the zero value of extinction coefficient (100 % transmission) shall be set by hardware or software as appropriate.

10.4 Less frequent calibrations

10.4.1 Operating heat flux meter calibration

At maximum intervals of 100 working hours, check the operating heat flux meter against the reference heat flux meter using one of the procedures described in Annex H, or the procedures described in ISO 14934-3. Comparisons shall be made at irradiance levels of (10, 25, 35, 50, 65, and 75) kW m⁻². The readings from the two meters shall agree to within \pm 2 %. If the operating heat flux meter is found to disagree with the reference meter by more than \pm 2 % the operating heat flux meter should be recalibrated and re-checked against the reference meter. If the operating heat flux meter cannot be brought to within a \pm 2 % agreement over the entire range the operating meter shall be replaced.

10.4.2 Linearity of heat release rate measurements

At maximum intervals of 100 working hours, with the instrument calibrated at 5 kW according to 10.2.4, perform a further calibration with a flow rate corresponding to 1 kW \pm 10 % and 3 kW \pm 10 %, using the basic procedure as described in 10.2.4. With the value for C from the 5 kW calibration, the measured heat release rate at 1 and 3 kW shall be within \pm 5 % of the set value.

10.4.3 Accuracy of calibration burner flow meter

The accuracy of the calibration burner flow meter shall be verified every 6 months or when the calibration factor determined according to $\underline{10.2.4}$ differs by more than 5 % from the value obtained during the first heat release rate calibration following the previous flow meter verification. To verify the accuracy of the flow meter, perform the burner calibration described in $\underline{10.2.4}$, with a reference flow meter in series with the operating flow meter. During the 3-min period of data collection, both flow meters shall agree to within \pm 3 %. If the difference between the two measurements exceeds \pm 3 %, the operating flow meter shall be re-calibrated as recommended by the manufacturer.

Alternatively, set the flow through the mass flow meter to a flow rate corresponding to $q_b = (5 \pm 0.5)$ kW. Then turn the methane off using a shut-off valve and replace the meter with a reference mass flow meter. Turn the methane on and measure the flow rate. Both flow meters shall agree to within \pm 3 %. If the difference between the two measurements exceeds \pm 3 %, the operating flow meter shall be recalibrated as recommended by the manufacturer.

11 Test procedure

11.1 General precautions

WARNING — So that suitable precautions are taken to safeguard health, the attention of all concerned in fire tests is drawn to the possibility that toxic or harmful gases can be evolved during exposure of test specimens.

The test procedures involve high temperatures and combustion processes. Therefore, hazards can exist such as burns or the ignition of extraneous objects or clothing. The operator shall use protective gloves for insertion and removal of test specimens. Neither the cone heater nor the associated fixtures shall be touched while hot except with the use of protective gloves. Care shall be taken never to touch the spark igniter which carries a substantial potential ($10~\rm kV$). The exhaust system of the apparatus shall be checked for proper operation before testing and shall discharge into a building exhaust system with adequate capacity. The possibility of the violent ejection of molten hot material or sharp fragments from some kinds of specimens when irradiated cannot totally be discounted and it is therefore essential that eye protection be worn.

11.2 Initial preparation

11.2.1 Check the CO_2 trap and the moisture traps. Replace the chemicals if necessary. Drain any accumulated water in the cold trap separation chamber. The normal operating temperature of the cold trap shall not exceed 4 °C.

If any of the traps or filters in the gas sampling line have been opened during the check, the gas sampling should be checked for leaks (with the sampling pump on), e.g. by introducing pure nitrogen, at the same flow rate and pressure as for the sample gases, from a nitrogen source connected as close as possible to the gas sampling ring probe. The oxygen analyser shall then read zero.

- **11.2.2** Adjust the distance between the base plate of the cone heater and the upper surface of the specimen as specified in 6.6 or 7.5.
- **11.2.3** Turn on power to the cone heater and the exhaust fan. Power to the gas analysers, weighing device and pressure transducer shall not be turned off on a daily basis.
- **11.2.4** Set an exhaust flow rate of (0.024 ± 0.002) m³ s⁻¹.
- **11.2.5** Perform the required calibration procedures specified in <u>10.2</u>. Put a thermal barrier on top of the weighing device (for example, an empty specimen holder with ceramic fibre blanket or water cooled radiation shield) in place during warm up and between tests to avoid excessive heat transmission to the weighing device.
- **11.2.6** The zero value of the extinction coefficient shall be set prior to every test.

11.3 Procedure

- **11.3.1** Start data collection. Collect one minute of baseline data. The standard scan interval is 5 s, unless a short burning time is anticipated (see <u>7.3</u>).
- **11.3.2** Insert the radiation shield in position (see 6.3). Remove the thermal barrier protecting the weighing device (see 11.2.5). Place the specimen holder and specimen, prepared according to Clause 8.3, on the weighing device.

The radiation shield shall be cooler than 100 °C immediately prior to the insertion.

11.3.3 Insert the spark plug and remove the radiation shield in the correct sequence according to the type of shield that is used, as described below.

For type a) shields (see <u>6.3</u>), remove the shield and start the test. Within 1s of removing the shield, insert and power the igniter.

For type b) shields (see <u>6.3</u>), remove the shield 15 s after the insertion and start the test. Within 1s of removing the shield, insert and power the igniter.

11.3.4 Record the times when flashing or transitory flaming occurs. When sustained flaming occurs, record the time, turn off the spark, and remove the spark igniter. If the flame extinguishes in less than 60 s after turning off the spark, re-insert the spark igniter and turn on the spark within 5 s, do not remove the spark until the entire test is completed. Report these events in the test report (Clause 13).

11.3.5 Collect all data until either:

- a) 32 min after the time to sustained flaming (the 32 min consist of a 30 min test period, and an additional 2 min post-test period to collect data that will be time-shifted). Data are processed to the time to sustained flaming plus 30 min.
- b) 30 min have elapsed and the specimen has not ignited;
- c) X_{02} returns to a value greater than the pre-test value minus 100 μ l/l of oxygen concentration for 10 min. The end of test is the beginning of the 10 min period
- d) the mass of the specimen is less than 0,1 g for 60 s. The end of test is the beginning of the 60 s period. whichever occurs first, but in any case, minimum test duration shall be 5 min. Observe and record physical changes to the sample such as melting, swelling, and cracking.
- **11.3.6** Remove specimen and specimen holder. Put a thermal barrier on top of the weighing device.
- 11.3.7 Three specimens shall be tested and reported as described in <u>Clause 13</u>. The 180 s mean heat release readings shall be compared for the three specimens. If any of these mean readings differ by more than 10 % from the arithmetic mean of the three readings, then a further set of three specimens shall be tested unless the mean value is less than 10 kW m^{-2} . In such cases, the arithmetic mean of the set of six readings shall be reported.

The smoke production rate test data shall not be discarded if piloted ignition does not occur, because the production rate data may have relevance under non-flaming conditions. The zero value of the extinction coefficient shall be verified prior to every test as part of the procedures specified in 11.2.

- NOTE 1 The test data have limited validity if the specimen melts sufficiently to overflow the sample holder, if explosive spalling occurs, or if the specimen swells excessively and touches the spark igniter or the heater base plate.
- NOTE 2 The heat release rate measurements described within this document, normally utilize piloted ignition. Separate non-standard tests may be conducted for research purposes without piloted ignition to evaluate smoke production rates under non-flaming conditions.

12 Calculations

12.1 General

The formulae from 12.2 to 12.5 assume that only O_2 is measured as indicated in the gas analysis system in Figure 6. Appropriate formulae for cases where additional gas analysis equipment (CO₂, CO and possibly H₂O) is used and CO₂ is not removed from the O₂ sampling lines can be found in Annex G. If CO₂ is removed from the O₂ sampling lines (even when CO₂ is separately measured), then the Formulae (6) to (8) shall be used.

The calculation for smoke obscuration is described in 12.6.

12.2 Calibration constant for oxygen consumption analysis

The heat release rate calibration specified in <u>10.2.4</u> shall be performed daily to check for the proper operation of the instrument and to compensate for minor changes in determination of mass flow. A calibration more than 5 % different from the previous one is not normal and suggests instrument malfunction. The calibration constant, *C*, is calculated using

$$C = \frac{\dot{q}_b}{(12,54 \times 10^3)(1,10)} \sqrt{\frac{T_e}{\Delta p}} \frac{1,105 - 1,5X_{O_2}}{X_{O_2}^0 - X_{O_2}}$$
 (6)

where \dot{q}_b corresponds to the rate of heat release (in kW) of the methane supplied (see 10.2.4), (12.54×10^3) kJ kg⁻¹ is $\Delta h_c/r_o$ for methane, 1,10 is the ratio of the molecular weights of oxygen and air.

12.3 Heat release rate

12.3.1 Prior to performing other calculations, calculate the oxygen analyser reading from the recorded analyser data and the delay time, t_d , using the following formula:

$$X_{o_2}(t) = X_{o_2}^1(t + t_{d}) \tag{7}$$

12.3.2 Calculate the heat release rate, $\dot{q}(t)$, from

$$\dot{q}(t) = (\Delta h_{\rm C} / r_{\rm o})(1,10)C\sqrt{\frac{\Delta p}{T_{\rm e}}} \cdot \frac{X_{\rm O_2}^0 - X_{\rm O_2}}{1,105 - 1,5X_{\rm O_2}}$$
(8)

where $\Delta h_{\rm C}/r_{\rm 0}$ for the specimen is taken as (13,1 × 10³) kJ kg⁻¹, unless a more accurate value is known, and X⁰02 is determined as the average of the oxygen analyser output measured during the 1-min baseline measurements.

12.3.3 Heat release per unit area can be obtained from

$$\dot{q}_{A}(t) = \dot{q}(t) / A_{S} \tag{9}$$

where A_s is the initially exposed area of the sample, 0,008 84 m². In case where the retainer frame is not used (see 8.3.2), A_s is 0,01 m⁻².

12.4 Exhaust duct flow rate

The mass flow rate, in kilo-grams per second, in the exhaust duct is given by

$$\dot{m}_{\rm e} = C \sqrt{\frac{\Delta p}{T_e}} \tag{10}$$

12.5 Mass loss rate

12.5.1 The mass loss rate, $-\dot{m}$, at each time interval can be calculated using the following five-point numerical differentiation formulae:

For the first scan (i = 0):

$$-[\dot{m}]_{i=0} = \frac{25m_0 - 48m_1 + 36m_2 - 16m_3 + 3m_4}{12\Delta t} \tag{11}$$

For the second scan (i = 1):

$$-[\dot{m}]_{i=1} = \frac{3m_0 + 10m_1 - 18m_2 + 6m_3 - m_4}{12\Delta t} \tag{12}$$

For any scan for which 1 < i < n-1 (where n is the total number of scans):

$$-\left[\dot{m}\right]_{i} = \frac{-m_{i-2} + 8m_{i-1} - 8m_{i+1} + m_{i+2}}{12\Delta t} \tag{13}$$

For the next to last scan (i = n-1):

$$-[\dot{m}]_{i=n-1} = \frac{-3m_n - 10m_{n-1} + 18m_{n-2} - 6m_{n-3} + m_{n-4}}{12\Delta t}$$
(14)

For the last scan (i = n):

$$-\left[\dot{m}\right]_{i=n} = \frac{-25m_n + 48m_{n-1} - 36m_{n-2} + 16m_{n-3} - 3m_{n-4}}{12\Delta t} \tag{15}$$

12.5.2 The mass loss rate which includes the "main" burning period, i.e. from 10 % of ultimate mass loss being lost to 90 %, is given by

$$\dot{m}_{A,10-90} = \frac{m_{10} - m_{90}}{t_{90} - t_{10}} \times \frac{1}{A_{S}} \tag{16}$$

where

$$\Delta m = m_{\rm S} - m_f$$

$$m_{10} = m_{\rm S} - 0.10 \Delta m$$

$$m_{90} = m_{\rm S} - 0.90 \,\Delta m$$

NOTE Formulae for the effective heat of combustion, $\Delta h_{c,eff}$, are given in Annex D.

12.6 Smoke obscuration

12.6.1 The extinction coefficient, *k*, is determined by the smoke meter electronics as follows:

$$k = \ln(I_0/I) L^{-1} \tag{17}$$

12.6.2 Smoke production rate per unit area of exposed specimen is given by:

$$P_{s,A} = A^{-1}k\dot{V}_{s} \tag{18}$$

The volumetric flow rate at the smoke meter, \dot{V}_s , is calculated from the mass flow rate measured with the orifice plate, \dot{m}_e , via

$$\dot{V}_{\rm s} = (\dot{m}_{\rm e} T_{\rm s}) (12, 2 \times 10^3 M)^{-1} \tag{19}$$

The value of T_s is obtained from the thermocouple described in <u>6.18</u> and not from the thermocouple associated with the orifice plate mass flow measurement.

If O_2 , CO_2 , CO and H_2O analysers are present; \dot{m}_e and M are obtained from Formulae (G.9) and (G.10) in Annex G.

12.6.3 The total smoke production per unit area of exposed specimen obtained during the non-flaming (pre-ignition) period of the test shall be calculated from:

$$S_{A,1} = A^{-1} \sum_{i=s}^{i=f} \dot{V}_s k \Delta t$$
 (20)

and the total smoke production per unit area of exposed specimen obtained during the flaming (postignition) period of the test shall be similarly calculated from:

$$S_{A,2} = A^{-1} \sum_{i=s}^{i=f} \dot{V}_s k \Delta t$$
 (21)

Where the values s and f for i refer to the start and the end of the time period over which the average is calculated. Thus, for the non-flaming phase, s refers to the start of the test and f to the time of the start of sustained flaming. For the flaming phase (if any); s corresponds to the time of the start of sustained flaming and f corresponds to the end of the flaming phase.

13 Test report

These test results relate only to the behaviour of the test specimens under the particular conditions of the test. They are not intended to be the sole criterion of assessing the potential fire hazard of the product in use.

The test report shall be as comprehensive as possible and shall include any observations made during the test and comments on any difficulties experienced during testing. The units for all measurements shall be clearly stated in the report. Units defined in Table 1 shall be used in the report.

The following essential information shall also be given in the test report:

- a) name and address of test laboratory;
- b) name and address of sponsor;
- c) name and address of manufacturer/supplier;
- d) date of the test;
- e) operator;
- f) trade name and specimen identification code or number;
- g) composition or generic identification;

- h) specimen thickness¹⁾, expressed in millimetres, and mass¹⁾, expressed in grams. With composites and assemblies a nominal thickness and density of each of the components shall be given, together with the apparent (overall) density of the whole;
- i) colour of the specimens;
- j) details of specimen preparation by the testing laboratory;
- k) specimen mounting, face tested, and any special mounting procedures (i.e. for intumescent specimen) that were used;
- l) orifice flow rate calibration constant *C*;
- m) irradiance¹⁾ expressed in kilowatts per square metre, and exhaust system flow rate¹⁾, expressed in cubic metres per second;
- n) number of replicate specimens tested under the same conditions (this shall be a minimum of three, except for exploratory testing.
- o) time to sustained flaming¹⁾, expressed in seconds;
- p) test duration¹⁾, i.e. the time between the start of the test and the end according to 11.3.5, expressed in seconds;
- q) heat release rate (per unit area), expressed in kilowatts per square metre, represented as a curve¹), recorded for the entire test;
- r) average values¹⁾ for the first 180 s ($\dot{q}_{A,180}$), and 300 s ($\dot{q}_{A,300}$), after ignition, or for other appropriate periods and peak1) \dot{q}_A ($\dot{q}_{A,max}$), values, expressed in kilowatts per square metre.

For specimens which do not show sustained flaming, report the above quantities tabulated for periods beginning with the next reading after the last negative heat release rate reading at the beginning of the test. Certain specimens do not show visible, sustained flaming, but do indicate nonzero heat release rate values. There will be negative readings, in general, since before the specimen starts burning the output is $0 \pm noise$.

Average heat release rate values shall be calculated using the trapezium rule for integration. For example, with a 5s data collection interval, $\dot{q}_{\rm A,180}$ is obtained as follows:

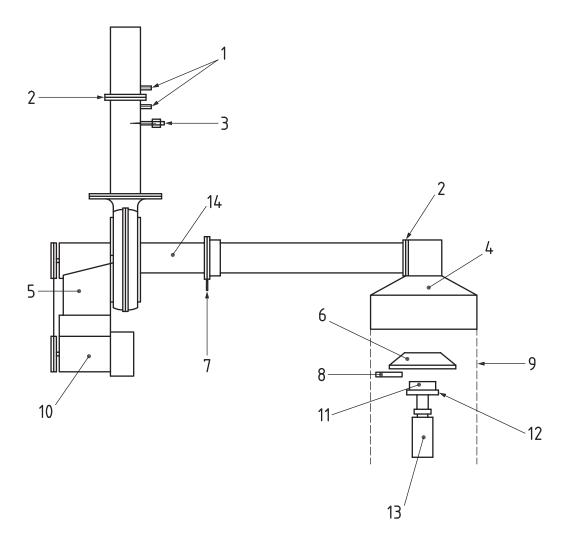
- 1) sum the rate of heat release rate values for 35 scans following the scan closest to ignition or the first scan after the last negative value; if the test is completed before the 180s period has elapsed, use the test average instead;
- 2) add half of the heat release rate measured at the scan closest to ignition or the first scan after the last negative value, and at the 36th scan after the scan closest to ignition or after the first scan after the last negative value;
- 3) multiply the sum obtained in step 2 by the scan interval (5) and divide by 180;
- s) total heat released by the specimen¹⁾, expressed in megajoules per square-metre. The total heat shall be computed beginning at the next reading after the last negative heat release rate reading occurred at the beginning of the test, and continuing until the final reading is recorded for the test.

The total heat release can also computed by using the trapezium rule to calculate integrated values. In this case, the first scan to be used is the one after the last negative heat release rate reading occurring at the beginning of the test;

t) mass¹⁾ at sustained flaming, m_s , and mass remaining after the test, m_f , both expressed in grams;

¹⁾ Report these items for each specimen.

- u) sample mass loss¹⁾, expressed in grams per square metre, and the average rate of specimen mass loss, \dot{m} , expressed in grams per square metre second (g m⁻² s⁻¹), computed over the period between ignition and the end of the test;
- v) average rate of specimen mass loss per unit area¹), $\dot{m}_{A,10-90}$, expressed in grams per square metre second (g m⁻² s⁻¹), computed over the period between 10 % and 90 % of mass loss;
- w) values determined in items o), p), r), s), t), u) and v), averaged for all replicates;
- x) additional observations¹⁾, such as transitory flaming or flashing;
- y) difficulties encountered in testing¹⁾, if any,
- z) total smoke production per unit area of exposed specimen over the non-flaming phase for every specimen $(S_{A,1})$,
- aa) total smoke production per unit area of exposed specimen over the flaming phase for every specimen $(S_{A,2})$.
- bb) total smoke production per unit area of exposed specimen for every specimen ($S_A = S_{A,1} + S_{A,2}$).
- cc) a graph showing the rate of smoke production per unit area as a function of time for every specimen ($P_{s,A}$ versus time), showing the time at first ignition.
- dd) the exposed surface area of the test specimen (*A*).

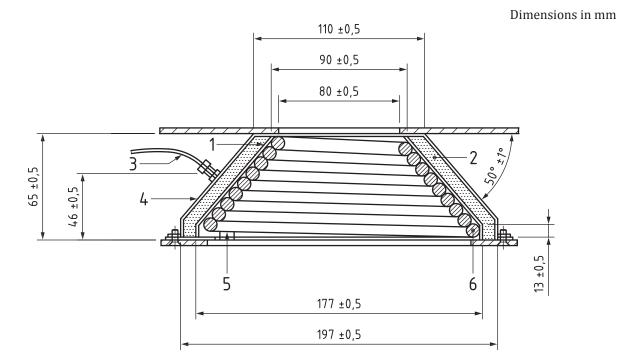


Key

- 1 pressure ports
- 2 orifice plate
- 3 thermocouple (located on stack centreline)
- 4 hood
- 5 blower
- 6 heater
- 7 gas sampling ring probe

- 8 spark plug
- 9 optional screens
- 10 blower motor
- 11 retainer frame and specimen
- 12 specimen holder
- 13 weighing device
- 14 smoke measurement section

Figure 1 — Apparatus



Key

- 1 inner shell
- 2 refractory fibre packing
- 3 thermocouple
- 4 outer shell
- 5 space block
- 6 heating element

Figure 2 — Cone heater

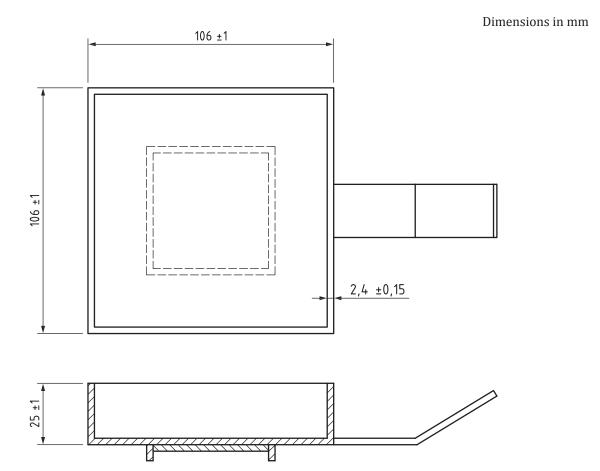
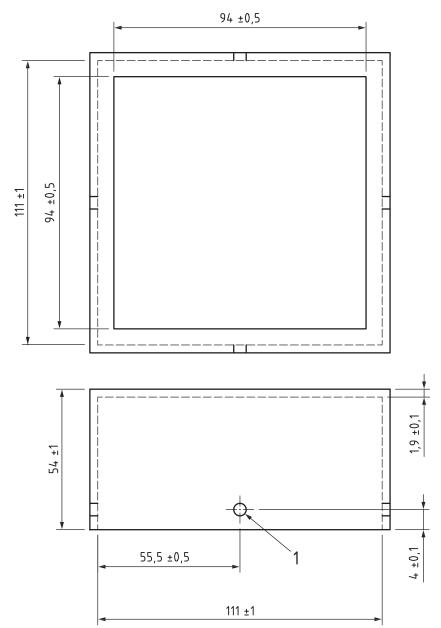


Figure 3 — Specimen holder

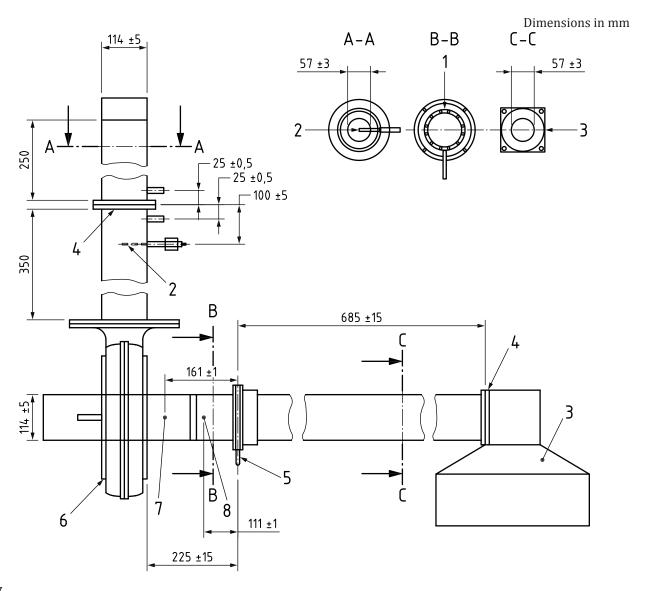
Dimensions in mm



Key

1 tapped holes in 4 places (M3 or 10 x 32 recommended)

Figure 4 — Retainer frame

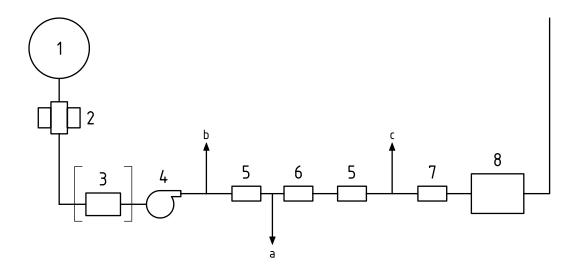


Key

- 1 gas sampling ring probe
- 2 thermocouple
- 3 hood
- 4 orifice plate

- 5 gas sampling ring probe (sample holes face blower)
- 6 fan
- 7 smoke thermocouple location
- 8 smoke meter location

Figure 5 — Exhaust system

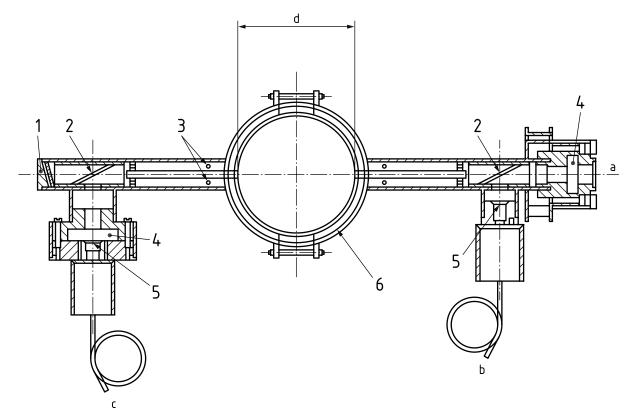


Key

- 1 gas sampling ring probe
- 2 particulate filter
- 3 optional cold trap
- 4 pump
- 5 moisture trap
- 6 optional CO₂ removal trap

- 7 flow controls
- 8 oxygen analyser
- $^{\rm a}$ To optional CO₂ and CO analysers.
- b Waste.
- c Alternative position for waste.

Figure 6 — Example for gas sampling and measurement system



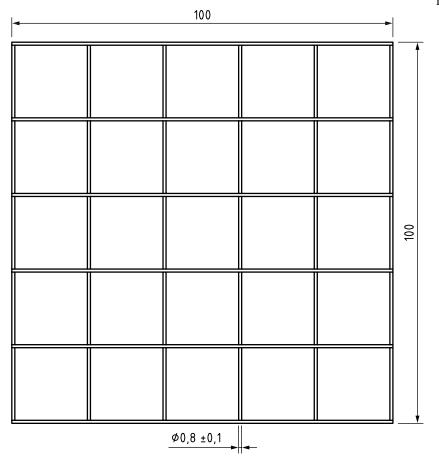
Key

- 1 cap
- 2 beam splitter
- 3 purge air orifices
- 4 filter slot
- 5 opal glass

- 6 ceramic fibre packing
- a He-Ne laser beam (0,5 mW).
- b To compensater detector.
- c To main detector.
- d Optical path.

Figure 7 — Cross section of typical smoke measuring arrangement

Dimensions in mm



 $Figure \ 8 - Fine \ wire \ grid \\$

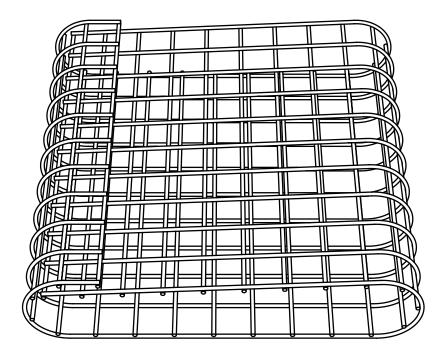


Figure 9 — Wire cage for materials that require testing under compression

Annex A

(informative)

Commentary and guidance notes for operators

A.1 Introduction

This annex aims to provide the test operator, and perhaps the user of the test results, with background information on the method, the apparatus and the data obtained.

A.2 Heat release rate measurements

- **A.2.1** Heat release rate is one of the most important variables in determining the hazard from a fire. In a typical fire, many items composed of many surfaces contribute to the development of a fire, thus making its evaluation quite complex. A determination should first be made when each separate surface will ignite, if at all. The size of the fire from any items already burning shall be known due to its contribution to the external irradiance on nearby items. Flame spread over each surface shall also be evaluated. The heat release rate from the whole surface is then determined knowing the heat release rate per unit area for a given irradiance, as a function of time, as evaluated using this bench scale test. The total fire output then involves a summation over all surfaces for all materials.
- **A.2.2** Factors which complicate the calculation of the heat output of a fire are:
- a) the different burning durations for each individual material involved;
- b) the geometry of each surface, and
- c) the burning behaviour of the material i.e. melting, dripping, or structural collapse.
- **A.2.3** The test method does not prescribe the irradiance levels. These should be determined separately for each product to be assessed. For given applications and products a comparison with some full-scale fires is generally necessary to determine the time period over which heat release is to be calculated.

For exploratory testing, it is recommended to use the spark igniter and an irradiance value of 35 kW m⁻² initially; in the absence of further specifications from the sponsor, tests at 25 kW m⁻², 35 kW m⁻² and 50 kW m⁻² are recommended. Results obtained then suggest whether additional testing at different irradiance levels is desirable.

The test results may not be statistically significant unless the irradiance used is substantially $(10 \, \text{kW m}^{-2})$ higher than the minimum irradiance level needed for sustained flaming to occur for that specimen.

A.3 Choice of operating principle

A.3.1 A number of apparatuses have been developed for measuring heat release rate. Traditionally, the simplest is a direct measurement of flow enthalpy from a chamber thermally lagged to present an adiabatic environment. A truly adiabatic apparatus, with the use of guard heaters, is possible, but would be prohibitively expensive. A combustion chamber that is insulated in a simpler manner leads to a significant under-measurement of the heat release, so that only an empirical calibration is possible. Furthermore, that calibration may be sensitive to the sootiness of the combustible. A more advanced scheme is an isothermal instrument, with the heat release rate taken to be that which shall be supplied by a substitution burner to maintain isothermal conditions. This scheme gives better results, however, its practical implementation is complex and costly.

A.3.2 It is difficult to measure heat directly without some loss. However, it is simple to contain the total products of combustion without loss and to measure the oxygen concentration in that stream. Heat release can be computed from such measurements using the oxygen consumption principle. This principle states that for most common combustibles an amount of heat equal to (13.1×10^3) kJ is released for each kilogram of oxygen consumed from the air stream. This quantity varies by about ± 5 % for most common combustibles. This principle forms the basis of the test method detailed in this part of ISO 5660. The method remains useful even if a significant fraction of the products become CO or soot rather than CO₂; in these cases, correction factors can be applied.

Excessively high CO concentrations which could result from a restricted oxygen supply, cannot occur under the normal operating conditions of this test method since oxygen intake is not restricted.

A.4 Heater design

- **A.4.1** Experience with various heat release rate measurement techniques suggests that for minimal errors in irradiance, the specimen should see only either a thermostatically controlled heater, or a water-cooled plate or open air. Nearby solid surfaces, if they are not temperature controlled, can rise in temperature due to specimen flame heating and can then act as further sources of radiation back to the specimen which can lead to errors. Furthermore, when oxygen consumption is used as the measurement principle, a gas-fired heater is not desirable because it can contribute a noisy baseline to oxygen readings, even though allowance can be made for its oxygen consumption.
- **A.4.2** The heater, in the shape of a truncated cone, initially developed for ISO 5657 has been modified to include higher irradiance, temperature control, flow streamline improvement and to be of a more rugged design. In the horizontal orientation, the conical shape approximately follows the fire plume contours while the central hole allows the stream to emerge without impacting on the heater. Air entrainment ensures the flames do not reach the sides of the cone.
- **A.4.3** Due to the shape of the heater, the apparatus is commonly referred to as the Cone calorimeter.

A.5 Pilot ignition

Ignition of test specimens in many apparatuses is achieved by a gas pilot. This however can present difficulties when assessing heat release due to its contribution to heat output, deterioration of orifices and sooting. Its design is also difficult since it should be centrally located, be resistant to extinguishment by draughts and fire retardants, and most importantly, additional heat should not be applied to the specimen. An electric spark is free of most of these difficulties and has therefore been adopted as the igniter. The spark igniter requires only occasional cleaning and adjustment of the electrodes.

A.6 Back face conditions

The heat losses through the back face of the specimen can have an influence on the burning rate near the end of its burning time. For reproducible measurements the loss through the back face should be standardized and this is achieved by using a layer of insulating material.

A.7 Light sources

The obscuration of light by an aerosol such as smoke occurs by two different phenomena: absorption and scattering. For highly detailed aerosol science studies, these two components can be measured individually. For fire safety concerns, however, normally only the total smoke obscuration is measured. Smoke obscuration is, by definition, the total attenuation due to both absorption and scattering.

Most early fire test methods such as the ASTM E662 [9] or the DIN test methods used a white-light source and some collimating optics, with a photodetector for sensing the light energy. However, from a theoretical point of view, polychromatic light is unsuitable for such measurement because Bouguer's

law is valid only for monochromatic light^[21]. Experimental studies ^[20] have confirmed theoretical predictions of the errors introduced by using white light.

Errors of this type can be avoided by the use of monochromatic radiation. A monochromatic light source can be created by filters or by monochromators. It is more convenient, however, to use a laser, which is an intrinsically monochromatic source. Readily available helium-neon lasers provide such monochromatic radiation at the red wavelength of 632,8 nm. A laser source has other advantages. As the source has sufficiently high intrinsic collimation, no lenses are needed. It also has a narrow beam which decreases errors due to multiple scattering.

A.8 Soot deposition on the optics

Traditional smoke measuring equipment generally has incorporated windows to exclude smoke from the optics. This has undesired consequences since during a test, soot is deposited on these windows. Consequently, the instrument sustains a drift of baseline and some approximate post-test corrections are needed. The availability of the very small diameter laser source allowed a different approach to be taken in the design of the apparatus^[23]. To avoid particle deposition on the optics, purging is provided by making use of the fact that the inside of the duct is at negative pressure with respect to the room outside. Furthermore, the beam tubes are purposely made long and narrow, so any ingressing particles will deposit on the tube walls instead of on the optics further out from the duct.

A.9 Photometer design

A conventional smoke photometer is a single-beam instrument. Thus, any changes in the source intensity due to power fluctuations, ageing, etc., are reflected directly as an error in the measured signal. Significantly better stability is obtained by a dual-beam design, whereby there are two photo detectors. One detector measures the smoke-attenuated light radiation, while the second detector only measures the source intensity, without any intervening smoke. By taking the ratio of these two signals, a high degree of stability is introduced into the measuring system. Such a dual-beam arrangement is provided in the present instrument^[24].

The laser photometer assembly is made of two pieces rigidly mounted to each other, but mechanically coupled to the exhaust duct only by resilient gaskets. This technique isolates the photometer from vibrations of the exhaust fan. The photometer can be used by either using an electronic circuit that takes the two detector signals and gives a final output directly in terms of the extinction coefficient k, or by taking the two detector signals into the data collection system and performing the arithmetic within the data reduction process.

The calibration of the photometer is done with the use of two optical filters of differing attenuation values which are inserted into a specially designed slot in the photometer. The use of two different values helps to verify that the calibration is linear. The photometer contains a second filter slot, located in front of the laser source. This filter slot is used to demonstrate that the two optical beams are in balance. If the detectors are properly matched and the system adjusted, then attenuating the source should not affect the final reading since both beams are equally attenuated.

The flow-through system used in the apparatus described in this part of ISO 5660 also minimizes other problems common to smoke measuring equipment; e.g. excessive wall losses and nonlinear effects due to soot overloading are endemic to closed-box smoke measurement systems. It was demonstrated by extensive comparisons conducted by the Fire Research Station [25] that the present arrangement is less prone to such errors.

A.10 Principles of smoke production rate measurement

The primary data from the photometer is expressed as the extinction coefficient, *k*. This is defined by the formula:

$$k = \ln\left(I_0/I\right) L^{-1} \tag{A.1}$$

where

- *I* is the attenuated beam intensity,
- I_0 is the beam intensity in the absence of smoke,
- *L* is the optical path length across the exhaust duct.

The smoke production rate, P_s , is calculated using the formula:

$$P_{\rm S} = k\dot{V}_{\rm S} \tag{A.2}$$

where \dot{V}_{c} is the volume flow rate.

The smoke production rate per unit area of exposed specimen, $P_{s,A}$, is calculated using the formula:

$$P_{S,A} = P_S A^{-1} \tag{A.3}$$

where *A* is the exposed surface area of the test specimen.

A.11 The calculation of the volume flow rate

The mass flow rate in the exhaust duct \dot{V}_s is calculated as described in <u>Clause 12</u>. However, in order to calculate the smoke production rate it is necessary to know the volume flow rate. This is derived from the mass flow rate using the formula:

$$\dot{V}_{s} = \dot{m}_{e} \rho^{-1} = \dot{m}_{e} \frac{T_{s}}{\rho_{0} T_{0}} \tag{A.4}$$

where ρ is the density of air at the photometer and is calculated using the formula:

$$\rho = 1{,}293 \text{ kg m}^{-3}(273{,}15 \text{ K/}T_{S}) \tag{A.5}$$

The density of air at standard temperature and pressure ρ_0 , is 1,293 kg m⁻³, T_S is the temperature in the duct close to the laser photometer, as determined by a thermocouple measurement at that location. No corrections are made for variations in pressure.

A.12 Smoke meter calibration using a calibration factor

Calibration using filters assumes that the system used to calibrate the filter is superior to the optical system of the smoke meter. The photodiodes used in the smoke meter specify a high degree of linearity. The optical density quoted for a commercially supplied filter is usually the average over a range of wavelengths and the value at the frequency of the laser may not be this average value. Therefore the use of a filter is better confined to use as a daily checking routine as to the functionality of the system rather than as a primary calibration. The user may therefore calibrate by checking the zero and 100 % transmission values and utilizing the linearity of the photodiodes.

If filters calibrated at the correct wavelength are used, the following routine may be followed.

Place a filter in the beam between the duct and the detector. Collect data for a period of 60 s. The measured calibration extinction coefficient, k_1 , is obtained from the formula:

$$k_1 = \ln(I_0/I)L^{-1}$$
 (A.6)

where L is the light path length through smoke. The correct value, k_2 , is given by the formula:

$$k_2 = (2,303 \, D')L^{-1}$$
 (A.7)

where D' is the optical density of the calibration filter.

A correction factor, k_2/k_1 , is calculated from these two values and is used to correct all subsequent measured k values, thus:

$$k = (k_2 / k_1) k_{\rm m}$$
 (A.8)

where k_m is the measured value.

Where a calibration factor, *F*, is used it is calculated as follows:

$$F = (k_2/k_1) L^{-1} \tag{A.9}$$

and subsequent *k* values are calculated using the formula:

$$k = F \ln \left(I_0 / I \right) \tag{A.10}$$

Annex B

(informative)

Supplementary calculations — Normalization to the mass loss rate of the specific extinction area of specimen

For the application of smoke data to fire models, it is sometimes desirable to report the data in terms of the yield of smoke per unit mass loss of specimen, independent of the apparatus flow conditions and specimen mass. To do this the specific extinction area is defined as the ratio of the extinction area of smoke to the mass loss of the specimen that is associated with the production of that smoke:

$$\sigma = k\dot{V}_s \Delta m^{-1} t \tag{B.1}$$

where

 σ is the specific extinction area, in square metres per kilogram;

k is the extinction coefficient, expressed per metre;

 $\dot{V}_{\rm s}$ is the volume flow rate of smoke at the point of measurement in a specified period in m³ s⁻¹.

 Δm is the specimen mass loss in a specified period, in kilograms;

t is the period of time for Δm , in seconds.

At any given time during the flaming phase of a test the specific extinction area can be calculated by dividing the smoke production rate by the mass loss rate:

$$\sigma = k\dot{V}_{s} \left(-\dot{m}\right)^{-1} \tag{B.2}$$

However, this formula should not be used if the mass loss rate factor is zero or less.

Calculation of the mass loss rate, \dot{m} , is described in 12.5.

The average specific extinction area over the flaming phase of the test is given by:

$$\bar{\sigma}_f = \left(m_{ig} - m_f\right)^{-1} \sum_{i=t_{ig}} \dot{V}_s k \Delta t \tag{B.3}$$

where

 m_{ig} is the specimen mass at ignition

 $m_{\rm f}$ is the specimen mass at the end of the test.

The reported variables would be

- a) $\bar{\sigma}_f$ over the flaming phase for every specimen, and
- b) a graph of σ as a function of time for every specimen

The average specific extinction area is a useful parameter in fire modelling because it is not sensitive to the scale of the fire. Thus, a value of σ obtained from a bench-scale test will be close to what is actually realized in a fire, provided that the combustion conditions are similar.

Additional information on smoke variables and their usage is given in references[11] and [26].

NOTE For materials containing absorbed water or molecularly bound water, mass loss measured will not fully represent mass lost by combustion.

Annex C (informative)

Resolution, precision and bias

C.1 Resolution

Methane calibration studies have shown typical fluctuations (due mainly to the turbulence of the flame itself) of ± 1,5 % in heat release rate with a linearity to within 5 % over a range of 1 kW to 12 kW, and within 2 % over a range of 5 to 12 kW. Calibrations with other gases show similar results. Calibration gases can be delivered to the burner at a steady rate. The uniformity of solid-fuel combustion, however, is governed by the pyrolysis at the surface, which can under some circumstances show substantial fluctuations. For instance, the fluctuations for poly (methyl methacrylate) are typically greater than for wood products. With solid materials, therefore, the resolution is determined by the specimen pyrolysis process, rather than by instrument limits.

C.2 Speed of response

The limits to the speed of response of any technique for measuring heat release rate are set by the slowest responding element. With this method this is the oxygen analyser. Response times of the pressure transducer and thermocouple are usually considerably faster.

C.3 Precision

The repeatability r and reproducibility R in <u>C.3</u> and <u>C.4</u> were calculated according to ISO 5725:1986 (now withdrawn) which was valid when the interlaboratory trials were conducted.

NOTE The current version of ISO 5725-1 reports r and R as $1 \times$ the relevant standard deviation, rather than $2.8 \times$ the standard deviation.

A set of interlaboratory trials was conducted by ISO/TC 92/SC 1/WG 5. The protocol used was functionally the same as described in this part of ISO 5660. The materials tested in these trials were: 25 mm black PMMA (ρ = 1 180 kg/m³), 30 mm rigid polyurethane foam (ρ = 33 kg/m³), 13 mm particle board (ρ = 640 kg/m³), 3 mm hardboard (ρ = 1 010 kg/m³),10 mm gypsum board (ρ = 1 110 kg/m³) and 10 mm fire-retardant-treated particle board (ρ = 750 kg/m³). Three replicates of each material were tested in two orientations (horizontal and vertical) and at two irradiance levels (25 kW m⁻² and 50 kW m⁻²) by six to eight laboratories.

Data from these trials were supplemented by data developed during an analogous set of trials conducted by ASTM E05 SC 21 TG 60, again using functionally the same protocol and identical irradiance levels, orientations and number of replicates. Since the findings for r and R in the ASTM trials showed generally similar trends, the data were analysed as a combined data set. ASTM data were excluded in the one instance (i.e. for $\dot{q}_{\rm A,180}$) where instructions to the laboratories differed. Six laboratories tested the following materials: 6mm fire-retardant-treated ABS (ρ = 325 kg/m³), 12 mm particle board (ρ = 640 kg/m³), 6 mm Black PMMA (ρ = 1 180 kg/m³), 6 mm Polyethylene (ρ = 800 kg/m³), 6 mm PVC (ρ = 1 340 kg/m³) and 25 mm rigid polyisocyanurate foam (ρ = 28 kg/m³).

Values for repeatability r and reproducibility R at the 95 % confidence level were calculated for the complete data set according to ISO 5725:1986 for five variables. Such values for r and R are equal to 2,8 × the appropriate standard deviation and include test results identified as "stragglers". The variables were chosen as being representative for the test results: $t_{\rm ig}$, $\dot{q}_{\rm A,max}$, $\dot{q}_{\rm A,180}$, Q_{Atot} and $\Delta h_{\rm c,eff}$. A linear regression model (Formula II in ISO 5725:1986) was used to describe r and R as a function of the mean

over all replicates and over all laboratories for each of the five aforementioned variables. The regression formulae are given below. The range of mean values over which the fit was obtained is also indicated.

The results for t_{ig} in the range of 5 s to 150 s were:

$$r = 4.1 + 0.125 t_{ig}$$
 (C.1)

$$R = 7.4 + 0.220 t_{ig}$$
 (C.2)

The results for $\dot{q}_{\rm A.max}$ in the range of 70 kW m⁻² to 1120 kW m⁻² were:

$$r = 13.3 + 0.131 \ \dot{q}_{A,\text{max}}$$
 (C.3)

$$R = 60.4 + 0.141 \dot{q}_{A,\text{max}} \tag{C.4}$$

The results for $\dot{q}_{\rm A~180}$ in the range of 70 kW m⁻² to 870 kW m⁻² were:

$$r = 23.3 + 0.037 \ \dot{q}_{A.180}$$
 (C.5)

$$R = 25.5 + 0.151 \ \dot{q}_{A.180} \tag{C.6}$$

The results for $Q_{A,tot}$ in the range of 5 MJ/m² to 720 MJ/m² were:

$$r = 7.4 + 0.068 Q_{A,tot}$$
 (C.7)

$$R = 11.8 + 0.088 Q_{A,tot}$$
 (C.8)

The results for $\Delta h_{\text{c.eff.}}$ in the range of 7 kJ/g to 40 kJ/g were:

$$r = 1,23 + 0,050 \,\Delta h_{\text{c.eff}}$$
 (C.9)

$$R = 2,42 + 0,055 \,\Delta h_{c,eff} \tag{C.10}$$

The meaning of these formulae is best illustrated by means of an example. Suppose a laboratory tests a single sample of a certain material and determines that the time to ignition is 100 s. If the same laboratory now conducts a second test on the same material, the value of r is evaluated as:

$$r = 4.1 + 0.125 \times 100 = 17 \text{ s}$$

Then the probability is 95 % that the result of the second test will fall between 83 s and 117 s. Suppose now that the same material is tested by a different laboratory. The value of *R* is evaluated as:

$$R = 7.4 + 0.220 \times 100 = 29 \text{ s}$$

Then the probability is 95 % that the results from the test at that laboratory will fall between 71 s and 129 s.

C.4 Precision (test procedures for materials that intumesce or deform)

A set of interlaboratory trials on materials that intumesce or deform when subjected to heat was conducted by ISO/TC 61/SC 4/WG3. The protocol used was functionally the same as described in this standard, with a distance between sample surface and heater base plate of 60 mm (instead of the standard 25 mm) as specified in Clause 7.5. The materials tested in these trials were: 9,6 mm black PMMA, 4 mm PVC, 3 mm

fire-retardant polypropylene, 5,8 mm polycarbonate, and 7,8 mm polycarbonate. Three replicates of each material were tested in the horizontal orientation, and at 50 kW m^{-2} by 10 laboratories.

Values for repeatability r and reproducibility R at the 95 % confidence level were calculated according to ISO 5725:1986 for three variables; $t_{\rm ig}$, $\dot{q}_{\rm A,max}$ and $Q_{\rm A,tot}$. A linear regression model (Formula II in

ISO 5725:1986) was used to describe r and R as function of the mean over all replicates and over all laboratories for each of the three aforementioned variables. The regression formulae are given below. The range of mean values over which the fit was obtained is also indicated.

The results for t_{ig} in the range of 27 s to 167 s were:

$$r = 2.3 + 0.255 t_{ig}$$
 (C.11)

$$R = 2.3 + 0.652 t_{ig}$$
 (C.12)

The results for $\,\dot{q}_{\rm A.max}\,$ in the range of 83 kW m⁻² to 855 kW m⁻² were:

$$r = 36.6 + 0.064 \dot{q}_{A,max}$$
 (C.13)

$$R = 36.6 + 0.330 \ \dot{q}_{\text{A max}}$$
 (C.14)

The results for $Q_{A,tot}$ in the range of 27 MJ/m² to 319 MJ/m² were:

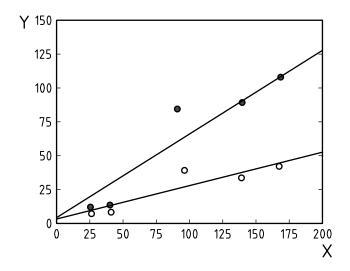
$$r = 15.5 + 0.008 \, Q_{A,tot}$$
 (C.15)

$$R = 15.5 + 0.125 \, Q_{A,tot}$$
 (C.16)

A comparison of Formulae (C.1) and (C.2) to (C.11) and (C.12) reveals that repeatability and reproducibility of ignition time is worsened by increasing the distance between the sample surface and the heater base plate to 60 mm. The repeatability of the remaining two variables does not seem to be affected [see Formulae (C.3) and (C.7) vs. (C.13) and (C.15)], but reproducibility is slightly worse for the 60 mm distance [see Formulae (C.4) and (C.8) vs. (C.14) and (C.16)].

C.5 Bias in heat release measurement

For solid specimens of unknown chemical composition, as used in building materials, furnishings, etc it has been documented that the use of the oxygen consumption standard value of $\Delta h_c/r_o$ = (13,1 × 10³) kJ/kg oxygen, results in an expected error band of ± 5 %. For homogeneous materials with only a single pyrolysis mechanism, this uncertainty can be reduced by determining Δh_c from oxygen bomb measurements and r_o from ultimate elemental analysis. For most testing, this is not practical since specimens are usually composites, non-homogeneous or exhibit several degradation reactions. For reference materials, however, careful determination of $\Delta h_c/r_o$ can make this source of uncertainty substantially less. See Figures C.1, C.2 and C.3.



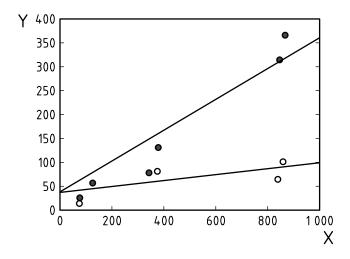
Key

X mean ignition time (s)

o r = 2.3 + 0.26 m

• R = 2.3 + 0.55 m

Figure C.1 — Values of r and R of t_{ig} for intumescent material



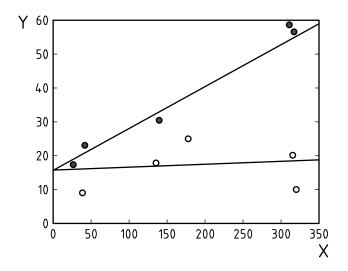
Key

X mean peak heat release rate

o r = 37 + 0.064 m

• R = 37 + 0.33 m

Figure C.2 — Values of r and R of \dot{q} $_{\rm A,max}$ for intumescent material



Key

- X mean total heat release rate (MJ m⁻²)
- o r = 1 + 0.008 m
- R = 16 + 0.133 m

Figure C.3 — Values of r and R of $Q_{A,tot}$ for intumescent material

C.6 Smoke precision

A series of interlaboratory tests for this part of ISO 5660 was carried out, among seven laboratories, on five specimens simulating upholstered furniture composites in the European fire research programme project of CBUF (Combustion Behaviour of Upholstered Furniture). In these tests, specific extinction area ($m^2 kg^{-1}$), which is based on the measurement of extinction coefficient of smoke and mass loss of specimen during the tests, was obtained in addition to heat release data. This parameter is explained in Annex B. Although this parameter is specified as an informative parameter, the results of the interlaboratory tests give precision data on smoke generation measurement method.

<u>Table C.1</u> presents the contents of the specimen which are combination of materials of upholstered furniture.

Table C.1 — Combinations of materials of upholstered furniture

Combination 1.	Back-coated acrylic fabric, 546 g m $^{-2}$, non-fire retarded high resilient polyurethane foam, 21 kg m $^{-3}$
Combination 2.	Fire retarded cotton fabric 422 g m ⁻² , combustion modified high resilient foam, 30 kg m ³
Combination 3.	Polypropylene fabric, 264 g m ⁻² , non-fire retarded polyurethane foam, 21 kg m ⁻³
Combination 4.	Wool fabric, 432 g m ⁻² , combustion modified high resilient foam, 30 kg m ⁻³
Combination 5.	Same as combination 1 but includes Kevlar interliner, 65 g m ⁻²

Table C.2 presents the data for repeatability r and reproducibility R as well as average value m. The analysis was carried out according to ISO 5725:1986 which was valid when the tests were conducted.

Table C.2 — Repeatability and repoducibility of specific extinction area (m²/kg)

Specimen	Laboratories reporting	m	r	R	
Combination 1:	6	399	93	366	
Combination 2:	5	108	60	076	
Combination 3:	6	499	91	112	
Combination 4:	6	241	27	056	
Combination 5:	5	341	93	333	

A linear regression model specified in ISO 5725:1986 can be used to describe r and R as functions of the mean. The following formulae are obtained from the data in Table C.2.

$$r = 28,83 + 0,14 m$$
 (C.17)

$$R = 15,03 + 0,56 m ag{C.18}$$

C.7 Bias in Smoke production measurement

The bias of this test method is unknown.

Annex D

(informative)

Mass loss rate and effective heat of combustion

D.1 Effective heat of combustion

The effective heat of combustion is a constant during combustion of homogeneous specimens having only a single mode of degradation and is less than the value of the theoretical net heat of combustion. Examples of a material with a single mode of degradation and, therefore, a constant effective heat of combustion include most organic liquids. Cellulosic products, by contrast, typically show more than one mode of degradation and a varying effective heat of combustion. For materials having more than one mode of degradation, or for composites or non-homogeneous materials, the effective heat of combustion is not necessarily constant. Effective heat of combustion and mass loss rate can be used to provide additional information on the fire behaviour of materials.

NOTE For materials containing absorbed water or molecularly bound water, mass loss measured will not fully represent heat of combustion.

D.2 Symbols

 $\Delta h_{c.eff}$ effective net heat of combustion, is expressed in megajoules per kilogram (MJ/kg)

D.3 Calculation

The mass loss rate, $-\dot{m}$, computed for each time interval starting from time of ignition (see 12.5.1), can be used to determine a time-varying value of the effective heat of combustion:

$$\Delta h_{\text{c,eff}} = \frac{\dot{q}(t)}{-\dot{m}} \tag{D.1}$$

As the mass loss rate requires numerical differentiation for its determination and is, therefore, noisier than measurements obtained directly from instrument readings, it is better to calculate average values of $\Delta h_{c,eff}$. To obtain such averages the numerator and denominator in Formula (D.1) shall be averaged separately rather than calculating the average of the ratio. For instance $\Delta h_{c,eff}$ averaged over the whole test is obtained as:

$$\Delta h_{\text{c,eff}} = \frac{\sum \dot{q}(t)\Delta t}{m_s - m_f} \tag{D.2}$$

The summation is taken over the entire test length from the time of ignition.

Annex E (informative)

Testing in the vertical orientation

E.1 Introduction

The normative sections of this standard are concerned with testing in the horizontal orientation only. This is applicable even to specimens, such as wall linings, where the end-use orientation of the product is vertical. The reason is that this test method does not represent a scale model of the full-scale product. Instead, the fundamental response of a specimen to specified external irradiance is tested. The total heating to the specimen is the sum of external irradiance plus the heat flux from the specimen's own flame. The heat flux from the specimen's own flames will be different in the two orientations. What shall be borne in mind is that there is no relationship between this flame flux for the bench-scale specimen, compared to the full-scale product. Instead, the relationship varies according to product application. The relationship between the bench-scale heat release rate and the one in full-scale should establish a test irradiance value which correctly accounts for the fact that the full-scale product is exposed to a different flame flux than is the bench-scale specimen.

The standard testing orientation is horizontal since, for most types of specimens, there are significantly fewer experimental problems due to specimen melting, dripping, or falling out. The reproducibility of ignition data are also better in this orientation, due to a wider column of pyrolysates present at the location of the spark gap. The vertical orientation is made available, because in certain diagnostic studies, it is more feasible to install optical pyrometers, specimen thermocouples, and other specialized instrumentation in that orientation. Minor modifications to the apparatus and test procedure are required for testing in the vertical orientation. These modifications are described in the following clauses.

E.2 Modifications to the apparatus

E.2.1 Cone-shaped radiant electric heater

For testing in the vertical orientation, the cone-shaped heater assembly shall be rotated over 90° , so that the base plate is vertical and parallel to the exposed face of the specimen.

E.2.2 Specimen holder

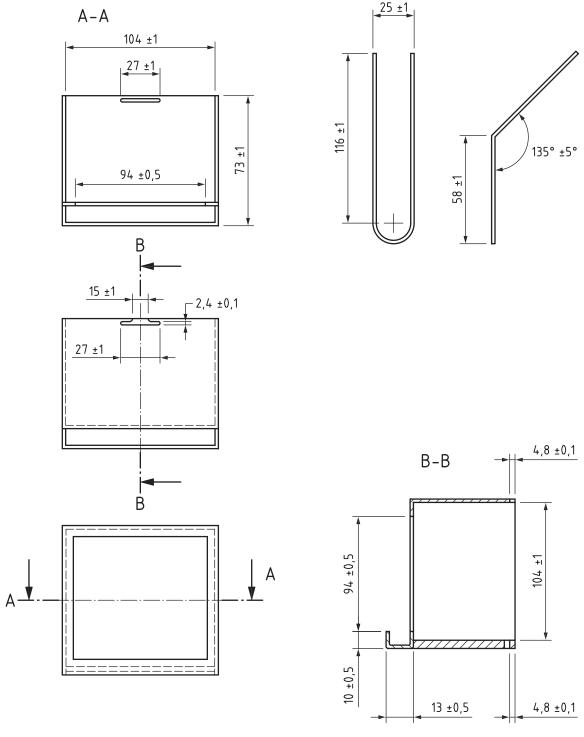
A different specimen holder than that described in <u>6.5</u> is required for testing in the vertical orientation. The vertical specimen holder is shown in <u>Figure E.1</u> and includes a small drip tray to contain a limited amount of molten material.

E.3 Specimen preparation

A specimen, wrapped in aluminium foil as described in 8.3.1, is installed in the vertical specimen holder by backing it with a layer of ceramic fibre blanket (nominal density 65 kg/m^3), the thickness of which depends on specimen thickness, but shall be at least 13 mm. A layer of rigid, refractory fibre millboard shall be placed behind the fibre blanket layer. The millboard thickness shall be such that the entire assembly is rigidly bound together once the retaining spring clip (see Figure E.1) is inserted behind the millboard. The cone heater height is set so the centre lines up with the specimen centre.

E.4 Heater calibration

The heater calibration in 10.2.5 shall be performed with the heater in the vertical orientation. The heat flux meter shall be positioned with its target facing the heater, at a location equivalent to the centre of the vertical specimen face.



NOTE Base plate is (4.8 ± 0.1) mm stainless steel. Other material is (1.59 ± 0.1) mm stainless steel.

Figure E.1 — Specimen holder for vertical orientation

E.5 Test procedure

The procedure for testing in the vertical orientation is largely identical to that for the horizontal orientation described in <u>Clause 11</u>. Prior to a test, the vertical specimen holder is positioned so that the exposed face of the specimen is parallel to and at a distance of 25 mm from the cone heater base plate. The spark plug described in $\underline{6.10}$ shall be positioned so that the gap is located in the specimen face plane and 5mm above the top of the holder.

Annex F (informative)

Calibration of the working heat flux meter

The inter-comparison of working and reference standard heat flux meters specified in <u>6.13</u>, may be made using the conical heater (<u>6.2</u>), with each heat flux meter mounted in turn in the calibration position. Care should be taken to allow the whole apparatus to attain thermal equilibrium. Alternatively, a specially built comparison apparatus may be used (for example that specified in BS 6809:1987).

The use of two, rather than one reference standard, provides a greater safeguard against change in sensitivity of the reference instruments.

Annex G (informative)

Calculation of heat release with additional gas analysis

G.1 General

The formulae in <u>clause 12</u> used to calculate heat release rate assume CO_2/CO are removed from the gas sample in a chemical scrubber before O_2 is measured, as indicated in <u>Figure 6</u>. Some laboratories are equipped to measure CO_2/CO in which case it is not necessary to remove the CO_2/CO from the O_2 line giving the advantage that the use of chemical scrubbing agent, which is costly and requires careful handling, can be avoided.

If formulae of this annex are used to obtain the heat release rate values, the response time(s) of the additional gas analyser(s) used shall closely match the response time of the oxygen analyser. If this requirement cannot be met, this annex shall not be used for obtaining the heat release rate. Silica gel shall not be used as the drying agent if a CO_2/CO analyser is used in the system.

In this annex, formulae are given which are to be used when CO_2/CO is measured but not scrubbed out of the sampling lines. Two cases are considered:

- in the first case, part of the dried and filtered sample stream is diverted into infrared CO₂ and CO analysers (see option in Figure 6);
- in the second case, a water vapour analyser is also added.

To avoid condensation, the measuring of H_2O concentration in the flow of combustion products requires a separate sampling system with heated filters, heated sampling lines, and heated analyser.

G.2 Symbols

The new symbols in <u>Table G.1</u> are used in this annex.

Table G1 — Symbols and their designations and units

Symbol	Designation	Unit
E	heat of combustion per unit mass of oxygen consumed (= $\Delta h_c / r_0$)	MJ/kg
E_{CO}	heat of combustion per unit mass of oxygen consumed for CO	MJ/kg
Н	relative air humidity	%
$M_{\rm a}$	molecular mass of air	kg/kmol
$M_{ m e}$	molecular mass of the combustion products	kg/kmol
$M_{\rm O2}$	molecular mass of the oxygen	kg/kmol
p	ambient pressure	Pa
$T_{\rm a}$	ambient temperature	K
t^1 d	delay time of the CO ₂ analyser	S
t^2 d	delay time of the CO analyser	S
t^3 d	delay time of the H ₂ O analyser	S
X^0 CO2	initial CO ₂ reading	(dimensionless)
X^0 CO	initial CO reading	(dimensionless)

Table G1 (continued)

Symbol	Designation	Unit
X ⁰ H2O	initial H ₂ O reading	(dimensionless)
<i>X</i> a ₀₂	ambient O ₂ mole fraction	(dimensionless)
X1 _{CO2}	CO ₂ reading before delay time correction	(dimensionless)
X1 _{CO}	CO reading before delay time correction	(dimensionless)
<i>X</i> 1 _{H20}	H ₂ O reading before delay time correction	(dimensionless)
$X_{\rm CO2}$	CO ₂ reading, mole fraction	(dimensionless)
$X_{\rm CO}$	CO reading, mole fraction	(dimensionless)
X _{H20}	H ₂ O reading, mole fraction	(dimensionless)
Φ	oxygen depletion factor	(dimensionless)

G.3 Case where CO₂ and CO are measured

G.3.1 Instrumentation

The CO_2 analyser shall be of the IR type and capable of measuring a range of at least 0 % to 10 % carbon dioxide. The linearity of the analyser shall be 1 % of full scale or better. The response time of the analyser shall be not more than 20 s (as measured in accordance with 10.1.5). The output from the analyser to the data acquisition system shall have a resolution of 100 μ l/l or better.

The CO analyser shall be of the IR type and capable of measuring a range of at least 0 % to 1 % carbon monoxide. The linearity of the analyser shall be 1 % of full scale or better. The response time of the analyser shall be not more than 20 s. The output from the analyser to the data acquisition system shall have a resolution of 10 μ l/l or better.

G.3.2 Carbon dioxide analyser adjustment

The carbon dioxide analyser shall be adjusted for zero and span each day on which tests are performed. The analyser output for carbon dioxide-free nitrogen gas shall be $(0,00 \pm 0,02)$ %. The span width shall be within 0,02 % $V_{\rm CO2}/V_{\rm air}$ of the width defined by the calibration gases used. A possible procedure to perform the adjustment is given below.

- a) For zeroing, feed the analyser with carbon dioxide-free nitrogen gas, with the same flow rate and pressure as for sample gases. When the analyser reaches equilibrium, adjust the analyser output to (0.00 ± 0.02) %.
- b) For span calibration a specified gas with a carbon dioxide content between 5 % and 10 % should be used. Feed the analyser with the gas, with the same flow rate and pressure as for sample gases. When the analyser reaches equilibrium, adjust the analyser output to the carbon dioxide content of the specified gas \pm 0,02 %.

G.3.3 Carbon monoxide analyser adjustment

The carbon monoxide analyser shall be adjusted for zero and span each day on which tests are performed. The analyser output for carbon monoxide-free nitrogen gas shall be (0,000 \pm 0,002) %. The span width shall be within 0,002 % $V_{\rm CO}/V_{\rm air}$ of the width defined by the calibration gases used. A possible procedure to perform the adjustment is given below.

- a) For zeroing, feed the analyser with carbon monoxide-free nitrogen gas, with the same flow rate and pressure as for sample gases. When the analyser reaches equilibrium, adjust the analyser output to $(0,000 \pm 0,002)$ %.
- b) For span calibration a specified gas with a carbon monoxide content between 0,5 % and 1,0 % should be used. Feed the analyser with the gas, with the same flow rate and pressure as for sample

gases. When the analyser reaches equilibrium, adjust the analyser output to the carbon monoxide content of the specified gas \pm 0,002 %.

G.3.4 Calculations

Just as for the oxygen analyser, measurements of CO_2 and CO shall be time shifted to take transport time in the sampling lines into account:

$$X_{02}(t) = X_{02}(t + t_d) \tag{G.1}$$

$$X_{\text{CO2}}(t) = X_{\text{CO2}}(t + t_d) \tag{G.2}$$

$$X_{\rm CO}(t) = X^{1}_{\rm CO}(t + t^{2}_{d}) \tag{G.3}$$

Here, the delay times t^1_d and t^2_d for the CO_2 and CO analysers respectively are usually different (smaller) than the delay time t_d for the O_2 analyser.

$$\dot{m}_e = C\sqrt{\frac{\Delta p}{T_e}} \tag{G.4}$$

The exhaust duct flow is calculated in the same way as in <u>clause 12.3</u>:

The heat release rate can now be determined from

$$\dot{q} = 1,10 \cdot E \cdot X_{O_2}^a \left[\frac{\Phi - \left(\frac{E_{CO} - E}{2E}\right)(1 - \Phi)X_{CO} / X_{O_2}}{(1 - \Phi) + 1,105 \Phi} \right] \dot{m}_e$$
 (G.5)

The oxygen depletion factor Φ follows from:

$$\Phi = \frac{X_{O_2}^0 (1 - X_{CO_2} - X_{CO}) - X_{O_2} (1 - X_{CO_2}^0)}{X_{O_2}^0 (1 - X_{CO_2} - X_{CO} - X_{O_2}^0)}$$
(G.6)

The ambient mole fraction of oxygen is:

$$X_{0_2}^a = (1 - X_{H_20}^0) X_{0_2}^0$$
 (G.7)

where

$$X_{H_20}^0 = \frac{H}{100 \cdot p} \exp\left[23, 2 - \frac{3816}{T_q - 46}\right]$$
 (G.8)

The second term in the numerator of the term in brackets in Formula (G.5) is a correction for incomplete combustion of some carbon to CO instead of CO_2 . In Cone calorimeter tests, X_{CO} is usually very small so that it can be neglected in Formulae (G.5) and (G.6). The practical implication of this is that a CO analyser will generally not result in a noticeable increase in accuracy of heat release rate measurements. Consequently Formulae (G.5) and (G.6) can be used even if no CO analyser is present, assuming X_{CO} is negligible and therefore set to 0.

G.4 Case where H₂O is also measured

In an open combustion system, such as used in this method, the flow rate of air entering the system cannot be measured directly but is inferred from the flow rate measured in the exhaust duct. An assumption is

required regarding the expansion due to combustion of the fraction of the air which is fully depleted of its oxygen. This expansion depends on the composition of the fuel and the actual stoichiometry of the combustion. A good average value for the volumetric expansion factor is 1,105, which is correct for methane.

This number is already incorporated within the formulae in 12.3.2 and Formula (G.5). For Cone calorimeter tests it can be assumed that over 99 % of the combustion products consist of O_2 , CO_2 , CO_3 , CO_4 , CO_5 , and non-reacting gases (which enter and leave the system chemically unaltered) denoted as CO_4 . If CO_5 is measured in the exhaust, this together with the CO_5 , CO_5 , and CO_6 measurements (all three referred to the dry gas) can be used to determine the expansion. The mass flow rate in the exhaust duct is then more accurately given by the following formula:

$$\dot{m}_e = \sqrt{\frac{M_e}{M_a}} C \sqrt{\frac{\Delta p}{T_e}} \tag{G.9}$$

In this formula M_a can be taken as 29 kg/kmol. The molecular weight of the combustion products M_e can be calculated from

$$M_e = \left[4.5 + (1 - X_{\text{H}_2\text{O}})(2.5 + X_{\text{O}_2} + 4X_{\text{CO}_2})\right] \cdot 4 \tag{G.10}$$

The heat release rate then follows from

$$\dot{q} = \frac{M_{\text{O}_2}}{M_e} \cdot E \cdot (1 - X_{\text{H}_2\text{O}}) \cdot X_{\text{O}_2}^a \cdot \left[\Phi - \left(\frac{\text{E}_{\text{CO}} - \text{E}}{2E} \right) (1 - \Phi) X_{\text{CO}} / X_{\text{O}_2} \right] \cdot \left[\frac{1 - X_{\text{O}_2} - X_{\text{CO}_2} - X_{\text{CO}_2}}{1 - X_{\text{O}_2}^0 - X_{\text{CO}_2}^0} \right] \cdot \dot{m}_e$$
(G.11)

It shall be noted that the H_2O readings shall be time shifted in a similar way as in Formulae (G.1) to (G.3) for the other analysers:

$$X_{\rm H_2O}(t) = X_{\rm H_2O}^1(t + t_d^3)$$
 (G.12)

Annex H (informative)

Calculation of Effective Critical Heat Flux for Ignition

H.1 General

Critical heat flux is for ignition is the minimum heat flux needed to sustain ignition. An effective Critical Heat Flux value can be determined empirically using the cone and igniter assembly section of the apparatus (or of the apparatus of this part of ISO 5660) by exposing specimens to different heat fluxes and measuring the ignition times for sustained flaming, initially observed. It is determined by trial and error with repeated tests in search of the heat flux for no ignition occurring within 15 min duration.

H.2 Procedure

The specimens shall be prepared as for a heat release test as in paragraph 8 but tested without a retainer frame.

Expose new specimens in each successive experiment to a different heat flux exposure and measure and record the time to sustained ignition of the vapours for each. The minimum heat flux is determined by trial and error. It shall first be determined to a coarse resolution of 5 kW m^{-2} , and then more finely to 1 kW m^{-2} .

Determine what is the lowest value of heat flux at which sustained ignition is achieved, and the highest value at which ignition is not achieved. The minimum heat flux for ignition is defined as the average between the lowest heat flux at which there is ignition, and the highest heat flux at which there is no ignition for 15 min. For example, if the specimen ignites at 30 kW m $^{-2}$ within 15 min, then repeat the same procedure at 25 kW m $^{-2}$, 20 kW m $^{-2}$, 15 kW m $^{-2}$ and 10 kW m $^{-2}$ (in that order) until there is no ignition for 15 min.

If test results show a crossover, in that the lowest heat flux at which ignition occurs is lower than the highest heat flux at which no ignition was found it will be necessary to carry out triplicate tests for each determination and average the results. The average of the (averaged) highest flux for non-ignition in 15 min and the averaged lowest heat fluxes for ignition within 15 min, is the reported value for the minimum heat flux for ignition.

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